EVALUATION AND OPTIMIZATION OF METAL MATRIX COMPOSITE STRIPS PRODUCED BY SINGLE ROLL CONTINUOUS STRIP CASTING
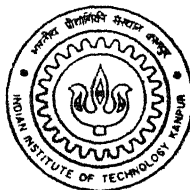
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May, 2000

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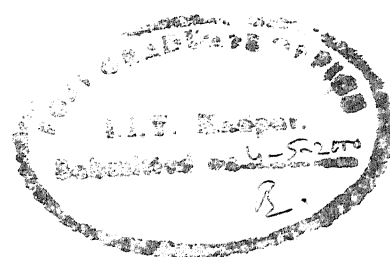## SINGLE ROLL CONTINUOUS STRIP CASTING

A thesis submitted
In partial fulfillment of the requirements
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Master of Technology

By
ROHIT KUMAR GUPTA

To the

DEPARTMENT OF MATERIALS AND METALLURGICAL
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MAY, 2000

CERTIFICATE

It is certified that the work contained in this thesis entitled *"Evaluation and optimization of MMC strips produced by Single Roll Continuous Strip Casting"* by **Rohit Kumar Gupta,** has been carried out under our supervision and that it has not been submitted elsewhere for any degree.

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Dedicated
To My
Prabhu, Guru
and
Parents

ACKNOWLEDGEMENT

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ABSTRACT

Composite materials are being used nowadays in every sphere of life. Among these composites, metal matrix composites with particulate reinforcement are finding applications in various fields. Conventional methods of composite strip production have lot of lacunae. In this context development of new technologies to produce composite strip becomes very important. The present investigation is an effort to apply the principle of vortex dispersion method (to prepare particle reinforced MMC melt) and to combine it with Single roll continuous strip casting technology, which produces composite strips in only one step. In this investigation Al-Si alloy and SiC ceramic particles are used as matrix and reinforcing material respectively.

The experimental investigation is essentially divided in two parts. First part consists of composite melt preparation and production of strip. During melt preparation wetting of particles by melt is improved by alloying with magnesium, preheating of particles and stirring of composite melt. Composite strips are produced for different sizes of reinforcing particle and with different wt. percentage of particle. To observe the effect of rotational speed of caster drum, strips at different rpm are also produced.

In the second part, the composite strips so produced are evaluated for their microstructure and mechanical properties. Microstructural studies are carried out using optical as well as electron microscope to examine the particulate distribution in the matrix , recovery of particle and internal quality of strips. Mechanical properties evaluation essentially included examination of tensile properties (tensile strength, 0.2% offset yield strength, % elongation), hardness and elevated temperature tensile properties (tensile strength and % elongation).

Processing parameters are optimized in every next set of experiment to get optimum combination of parameters.

It has been observed that size of the reinforcing particle directly affects the mechanical properties and distribution of particle in the matrix. Smaller size particle improves the strength and hardness of the composite. But in case of coarse particles (53-75μm) distribution and recovery is found better than small size

particles (<25μm). Better distribution of particle and increase in strength is also found at higher rpm of the caster drum. Composite strip shows better strength at elevated temperature (<250^{0}C) than the matrix alloy. Improvement in ductility of composites is observed at elevated temperature.

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CHAPTER 1

Introduction

Modern technology has placed severe demands on materials. One way to achieve the required quality is to combine two or more elemental materials to form composites, which can show excellent properties not achievable by any of the elemental materials acting alone. Desired combination of the useful properties can be obtained through proper selection of their constituent as well as methods of their synthesis.

Composites are non-equilibrium combination of matrices and reinforcing phases, where there are fewer thermodynamic restrictions on the relative volume fraction, shape and size of the later and wherein the constituents retain their individual identity in terms of property [1].

According to nature of the matrix, Composites can be classified as:
a.   Ceramic Matrix Composites (CMCs)
b.   Polymer Matrix Composites (PMCs)
c.   Metal Matrix Composites (MMCs)

On the basis of the nature, shape and size of the reinforcing phases, Composite materials are called:
a.   Particulate Composites
b.   Fiber Composites
c.   Whisker Composites
d.   Laminated Composites

This thesis is primarily concerned with Metal Matrix Particulate Composites. So this particular class of composite is discussed here in some detail.

1.1 METAL MATRIX COMPOSITES (MMCs) :

MMCs are engineered combination of two or more materials (one of which is metal) where tailored properties are achieved by systematic combinations of different

constituents. MMCs with particles in metal achieve combination of high strength and specific modulus with greater isotropy [1,2]. The use of these materials is being explored in many applications, including automobiles, aerospace and sporting goods. Some potential applications of particle reinforced Al/ SiC MMCs is listed in Table 1.1.

A variety of processes has been adopted to synthesize MMCs. These include:
- powder metallurgy methods,
- infiltration method,
- dispersion processes,
- spray deposition processes, etc.

Vortex liquid dispersion method is one of the inexpensive methods of composite melt preparation, which is used in this investigation. Brief description of these processes is presented in the second chapter on literature review.

1.2 COMPOSITE STRIP PRODUCTION:

Conventional method of MMC strip / sheet production involves the following step [16].
>Casting of ingots/billets
>Rolling of ingots/billets to produce strips
>Intermediate heat treatment
>Various finishing operations.

Since the Composites are more brittle than metal matrix, rejection rate of the rolled composite products due to crack formation is comparatively higher. Thus special precautions are necessary during the rolling operation making these quite expensive and energy extensive. As a result the total cost of production is high and productivity is low.

Development efforts in continuous casting have been directed mainly towards increase in productivity. For the last four decades several continuous strip casting techniques have been developed. Some continuous strip casting techniques are:
- chill block melt spinning,
- twin roll casting,

- melt drag process, etc.

Single roll continuous strip casting (SRCSC) is one of the newly developed techniques to produce near net shape continuous strip. Attempt is made to adapt this technique for production of MMCs strip in this investigation. Brief description of the above processes is given in the second chapter on literature review.

Table 1.1 Application of Al-Si alloy/SiC Composites [7-11].

Application	Specific properties
Compressive Vanes and Vacuum pump rotor.	Lightweight, wear resistance, low CTE.
Automobile pistons, engine parts	Light weight, high strength, wear resistance, low CTE, high thermal conductivity and stiffness
Propeller shaft	Light weight, high sp. Stiffness.
Shock absorber cylinder	Light weight, wear resistance, good thermal diffusion
Face of screw driver	Light wt., abrasion resistance.
Electronic racks	Wt. savings, superior isotropic stiffness.
Guidance components in place beryllium	Low costs, non-toxic, low CTE.
Brake components (disk brake rotors)	Wt. Savings, high thermal conductivity.
Electronic packaging	Low CTE, high stiffness, high thermal conductivity, light wt.
Heavy machinery in aerospace	Superior damping properties.
Some other applications include, Tennis rackets and golf goods, mountain bike frames, etc.	Light weight and durability.

1.3 OBJECTIVE AND PLAN OF THE PRESENT WORK :

Addition of second phase particles to aluminum based alloys (Al-Si, Al-Cu, Al-Si-Mg, Al-Zn) exhibited tremendous improvement in the mechanical, tribological and elevated temperature properties of the materials [12-14]. Reinforcement with SiC has undoubtedly received the greatest attention. Its attraction includes relatively low cost and ready availability, high modulus and strength. In this investigation Al-Si alloy and SiC particle are used as matrix and reinforcement respectively for composite development. The aim of the present work is to adapt the SRCSC method for production of MMCs strip. These strips are then evaluated for their microstructural and mechanical properties, to ensure that these strips are superior or at least as good as strips produced by other conventional techniques. Processing parameters are optimized for the above purpose.

This investigation can be broadly divided into two parts. The first part essentially deals with the preparation of particle reinforced metal matrix composite melt by vortex method and production of composite strips using single roll continuous strip casting (SRCSC) method.

In the second part the MMC strips so produced are evaluated for their microstructure and mechanical properties. Microstructure evaluation is primarily based on optical as well as electron microscope to examine the distribution of the ceramic particles in the composite strip and examination of porosity, inclusions etc. Mechanical property evaluation involves tensile testing of strips, hardness measurement and high temperature .tensile property measurements.

This thesis essentially consists of five chapters. This chapter on Introduction, is followed by chapter on Literature Review. The experimental procedure employed in this investigation is described in the third chapter. The results are presented and discussed in the fourth chapter. Conclusions of this investigation are presented in the chapter five. An appendix is given at the end of the thesis.

CHAPTER 2

Literature Review

This chapter contains three sections. In first section MMCs preparation and strip casting methods are presented. Phenomena on melt preparation and solidification of MMCs is discussed in section two. Review on strengthening and failure behavior of MMCs is presented in the last section of this chapter.

2.1 METHODS TO PRODUCE MMCs STRIPS :

2.1.1 MMCs preparation methods:

Techniques for MMCs preparation available are [15]:

1. Solid state processing methods: It includes Powder metallurgy method and diffusion bonding methods.
2. Liquid state processing methods: It includes composite melt preparation combined with casting processes.

A majority of the commercially viable applications are now produced by liquid state processing methods because of inherent advantages of this technique over solid-state techniques. Some of the advantages are: liquid metal is generally less expensive and easier to handle than powders, composite materials can be produced in a wide variety of shapes (using the methods of casting), suitable for various kinds of matrices and reinforcements.

Conversely, liquid state processing often suffers from lack of reproducibility as a result of incomplete control of the processing parameters and of undesirable chemical reactions at the interface between molten metal and reinforcement. Also, they are often limited to low melting point alloys.

Several liquid-state processing technologies currently being investigated and developed. Some important methods are described briefly in this section [15-17].

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2.1.1.1 Infiltration processes:

These processes involve holding a porous body of the reinforcing phase within a mold and infiltrating it with molten metal that flows through interstices to fill the pores and produce a composite.

2.1.1.2 Dispersion processes:

In these processes, the reinforcement is incorporated in loose form into the metal matrix. This method is used to prepare MMCs reinforced with discontinuous fibers or particles. Processes can be divided into two groups:

(a)   Compocasting method.

(b)   Vortex method.

In compocasting method reinforcement particles are added to a semi solid melt. An outstanding advantage of this method is its ability to incorporate materials, which are not wetted by the alloy melt. The non-wetting particles are dispersed by stirring action and are mechanically trapped by semi solid alloy. Processes like Settling, floatation, or agglomeration are also prevented by the presence of the semi solid alloy. But it has certain demerits; as well which limit its application. A special kind of stirrer capable of withstanding the corrosion and abrasion of the semi-solid melt is necessary, and a more stringent temperature control is required to maintain the melt in semi-solid state during stirring.

The problems associated with the compocasting method are eliminated to a large extent in the vortex method, which employs completely molten metals. Since vortex method is used in this thesis for melt preparation it is discussed in detail in experimental procedure chapter 3.

2.1.1.3 Spray Deposition processes:

In these processes, droplets of the molten metal are sprayed together with the reinforcing phase and collected on a substrate where metal solidification is completed. Alternatively, the reinforcement may be placed on the substrate where molten metal may be sprayed on to it.

2.1.1.4 In-Situ processes:

It is used for materials produced by solidification of polyphase alloys. Reinforced intermetallic alloys are produced by controlled solidification or by some other in-situ processes such as chemical reaction between a melt and solid or gaseous phases. Schematic diagrams for various processes are shown in Fig. 2.1.

2.1.2 Methods to produce MMCs Strip/Sheet:

This section presents an overview of the methods presently being used to produce MMC strips. These include:

♦ laminated metal composite technique,

♦ powder metallurgy method,

♦ continuous strip casting method.

These methods are described briefly in this section.

2.1.2.1 Laminated Metal Composite Technique:

LMCs are a unique form of composite material in which alternating metal or metal containing layers are bonded with discrete interfaces. Intermetallic matrices are reinforced with ductile metals in the form of particles, fibers, tubes and layers. These composites have been fabricated via pressure-aided consolidation of prealloyed intermetallic powders mixed with reinforcement. Some of the laminated metal composite techniques are briefly described below [18,19].

(a) Bonding technique starts with component materials in sheet or plate that are then solid state bonded at the interfaces. Adhesive bonding, reaction bonding, diffusion bonding and deformation bonding are some of the techniques that are applied nowadays in practice. Critical properties (such as fracture toughness, fatigue and impact behavior) are strongly influenced by local delaminations at interfaces, which limits its properties and use.

(b) In Deposition techniques, layers of component materials are formed sequentially by atomic or molecular scale transport of component materials. Sputtering, evaporation, chemical or physical vapor deposition (CVD or PVD) are some of the deposition techniques.

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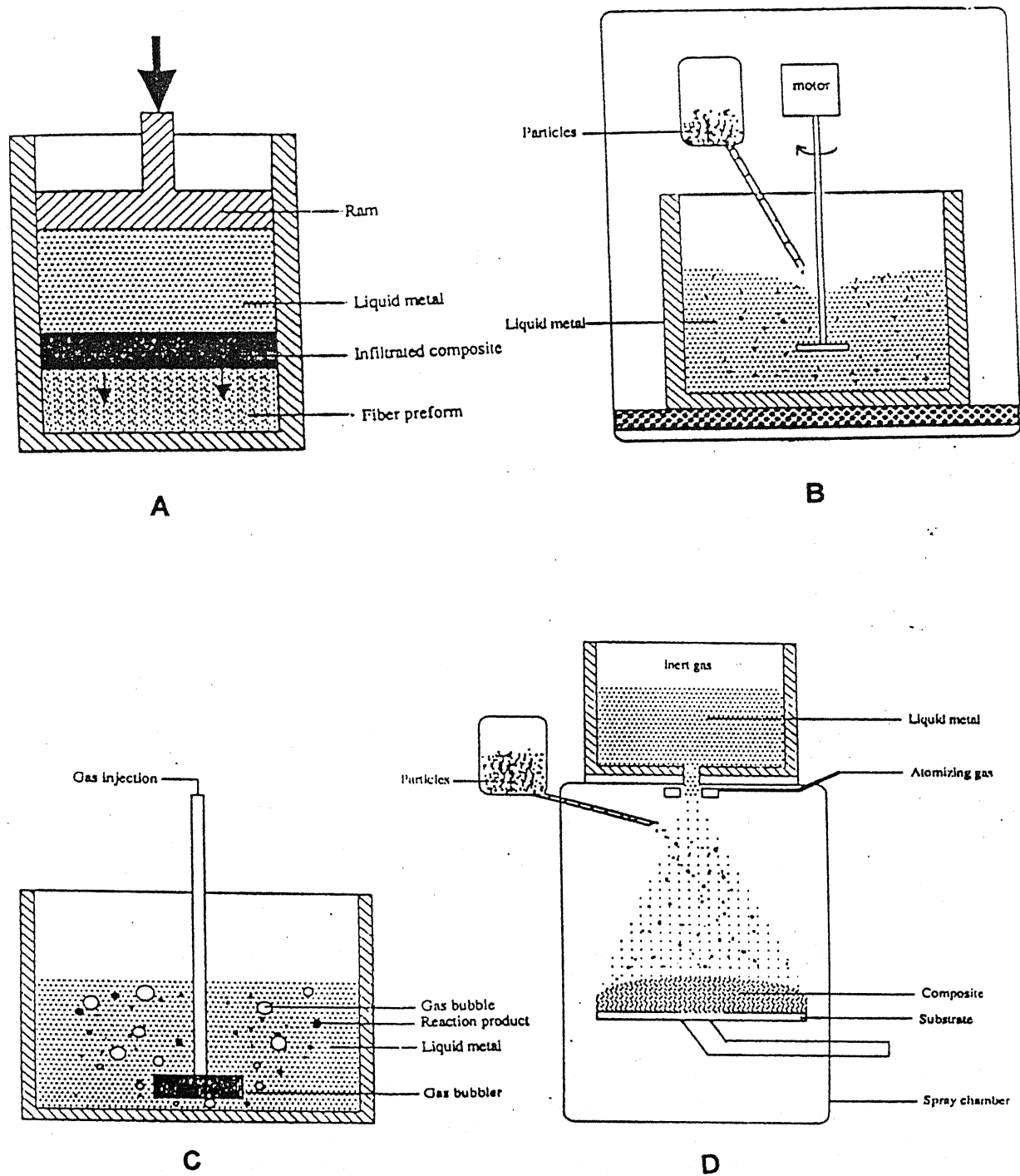
Fig. 2.1 : Schematic diagrams of various liquid-state processing
techniques [15-17]
A- Infiltration technique, B- Dispersion method, C- In-situ
method, D- spray deposition technique.

(c) <u>Spray forming techniques</u> involve direct deposition of molten metals of the component materials into a laminate form. Wu *et al.* spray deposited a multilayer laminate of 6061 and 6061-SiC, with a global SiC volume fraction of 15% by periodically injecting SiC particles into the atomized spray of the liquid Al 6061.

2.1.2.2 Powder Metallurgy Method:

In Powder Metallurgy methods finely divided powder particles are compacted and consolidated, which is further passed through roll to get strip / sheet of the desired thickness. Some of the P.M. techniques are [16]:

- Roll compaction
- Isostatic pressing

P.M. route requires more time, costly equipment and is more cumbersome and process expensive.

2.1.2.3 Continuous Strip Casting (Melt Spinning) Method [3-6]:

Despite their highly promising mechanical and thermal properties, MMCs have for a long time, been afforded only limited use in very specific applications. Shortcomings such as complex processing requirements and the high cost of the final product have presented the greatest barrier to their proliferation. Improvements in the reinforcement fabrication and composite processing techniques are therefore pivotal for increasing their commercial applicability. Significant efforts have been, and continue to be, devoted to this end with encouraging result; reinforced metals have begun to show their presence in large-scale commercial applications. To make it economical different techniques of continuous casting are used. Some new techniques of continuous strip casting are briefly given here.

1. Chill block melt spinning (CBMS): It involves the formation of a molten jet impinging on a rotating disk. The typical ribbon width during CMBS is about 3 mm and ribbon thickness varies between 20-200 µm depending on substrate velocity.

2. Twin roll continuous casting: This process was developed commercially by Hunter in 1950's. In this process the liquid jet impinges between the fast moving rolls and a solid strip can be produced directly from molten metal. Traditionally, industrial twin roll casters have been operated at casting gauges between 6 and 10 mm.

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3. Melt drag process: In this process metal is applied to the rotating drum through a nozzle, which gets removed by contact of meniscus with the rotating drum. By applying slot type nozzle wider tapes are formed and strip with widths upto 300 mm have been produced by this method. The thickness of the product depends, on metal flow rate, the melt temperature and peripheral speed of the drum.

Schematic diagrams of various 'continuous strip casting techniques' are shown in Fig. 2.2.

Based on the principle of the Melt drag process, Mehrotra & coworkers developed a Continuous Strip Caster. Since this method is used in this investigation, a detail description is given in experimental procedure, chapter 3.

2.2 PHENOMENA ON MELT PREPARATION AND SOLIDIFICATION OF MMCs:

2.2.1 Wetting of particle by liquid melt:

Wetting has very important role in MMC preparation, which determines the feasibility of particle incorporation in liquid melt. This is the subject of discussion of this section. The wettability of a solid by a liquid melt is represented by the solid-liquid contact angle θ. For wetting to take place the necessary condition is, contact angle must be less than 90^0. Contact angle and its relation with interfacial surface energies are given in appendix A 1.

2.2.1.1 Factors affecting wettability:

To analyze the phenomenon of wetting in detail, it is necessary to consider the factors that determine the extent of wetting [16,20-22]. These are:

- surface Tension
- adsorption
- work of adhesion
- presence of oxygen
- surface characteristics of reinforcement
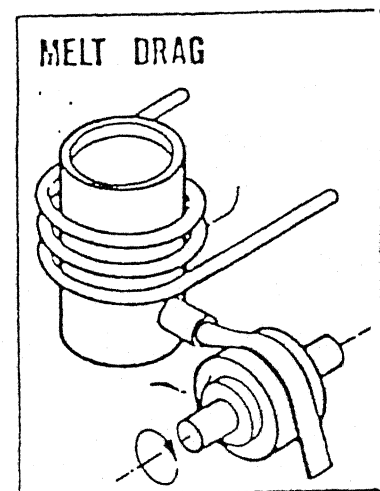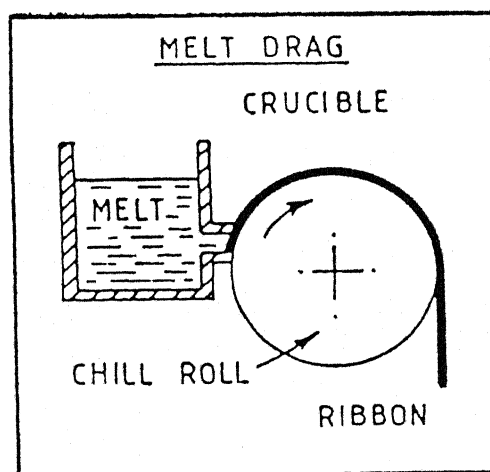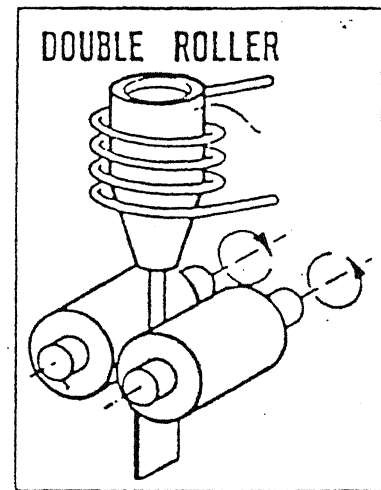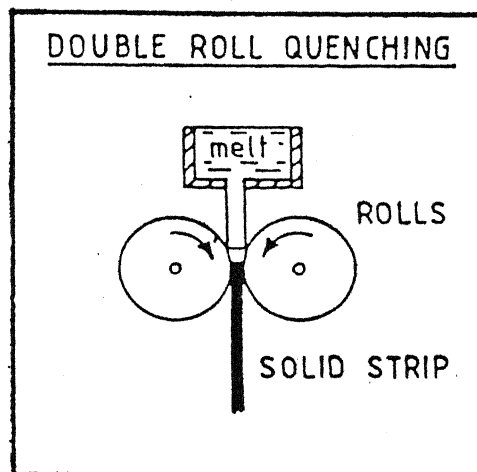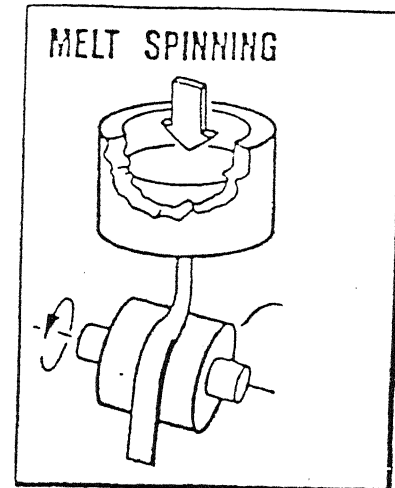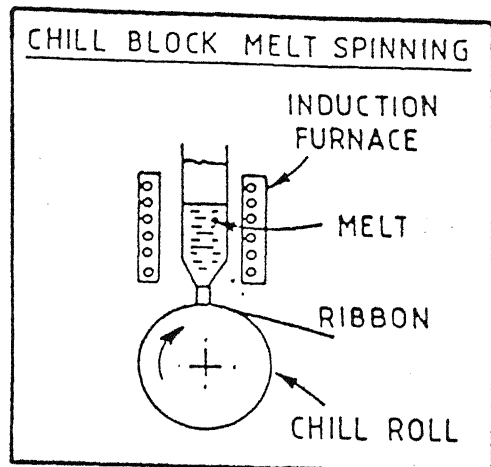
Fig. 2.2 : Schematic diagrams of various continuous strip casting techniques [3-6]

2.1.1.2 Approaches to improve wettability:

Attempts have been made to improve the wetting of particles by employing various techniques. These include:

(a)   coating of reinforcing particles

(b)   addition of alloying elements

(c)   preheating of ceramic particles

(d)   stirring of melt

(e)   temperature of melt and stirring

♦ **Coating of reinforcing particles:** The wettability is highest in the case of mutual solubility or formation of intermetallic compounds. These two phenomena changes the interfacial energies in a way so as to decrease the contact angle, thus, assisting better wetting between melt and particle [16].

Nickel, silver, copper and chromium coatings have been proposed for coating of particles. Coating element is selected according to surface characteristics of particle and matrix and their nature of interaction [20].

♦ **Addition of alloying elements:** Elements, which have a high affinity for oxygen, lower the interfacial energy of liquid metals with oxides. The most efficient alloying element reported to promote the wettability of the reinforcement is lithium. Addition of the surface active elements like Mg in Al melt, just prior to particle addition has also been employed in preparation of Al-graphite, Al-mica, Al-Al$_2$O$_3$, composites [23,25]. It has been found by Tafto *et al.* Mg has ability to generate vacuum by reaction with air, which improves wettability of particles, by Al alloys [24].

According to another observation made by Ohstu, effect of Ca or Mg is that, it accumulates in high concentrations in the vicinity of the surface of SiC particles, reducing the surface tension of Al and increasing the wetting properties thus reducing the dispersion time [7].

It has been observed that wetting of SiC with Al can be improved by addition of silicon in the melt or by using alloy of Al-Si [21]. This is due to the fact that prior alloying

prevents the formation of brittle Al_4C_3 phase at the interface, which is shown in section 2.2.2.2.

♦ **Preheating of particles:** The presence of contaminants on the ceramic particles is presumably responsible for non-wetting. Thermodynamically, SiC is very prone to oxidation and the surface of SiC particles are always covered by a stable film of SiO_2. Wetting of SiC thus amounts to wetting of SiO_2 surface. It has therefore been proposed that, in order to provide good wettablity it should be heated to a high temperature to burn away contaminants and to form a stable clean SiO_2 surface film. Preheating of particles also removes moisture, if any and avoids the agglomeration of particles after addition. These directly affect the interfacial energies thereby assisting in improved wetting.

From the experimental observation of Rohatgi *et al.* shown in Table 2.1 it can be inferred that the optimum combination of preheating temperature and time is 900°C and one hour. It applies to SiC particles and ellite clay as well. The temperature and duration of heat treatment are different for different Metal matrix reinforcement systems [16,20,27].

Table 2.1: Effect of preheat treatment temperature and time on recovery of particles [27].

Particles	Wt. % added	Preheating Temperature (°C)	Preheating Time (hr)	% recovery
200 µm Al_2O_3	3	300	24	Nil
200 µm Al_2O_3	3	700	4	Nil
200 µm Al_2O_3	3	900	1	96
200 µm Al_2O_3	3	900	0.5	70
60 µm Al_2O_3	3	900	1	83
< 53 µm SiC	3	900	1	33
< 53 µm illite clay	3	900	1	50

+ **Stirring of Melt:** Stirring of melt during and after addition of the reinforcing particles improves wettability of particles by the melt [16]. Impeller rpm coupled with blade angle becomes the controlling variable in achieving a good mix. A marked increase in rpm is required with increase in particle density to achieve complete dispersion [28].

+ **Temperature of Melt and stirring time:** Temperature of the melt and the time for which it is held at this temperature during preparation of melt, influence the wetting characteristics considerably. Brennam *et. al.* observed that at low partial pressure of oxygen the sapphire surface is changed to an oxygen deficient spinel type structure containing AlO, which reacts with clean molten Al to form a volatile species, Al_2O. This is a kinetic process and occurs readily above 900-1000 °C. This phenomenon increases with increase in vacuum [23,29]. Variation of contact angle with temperature and time is seen in Fig 2.3.
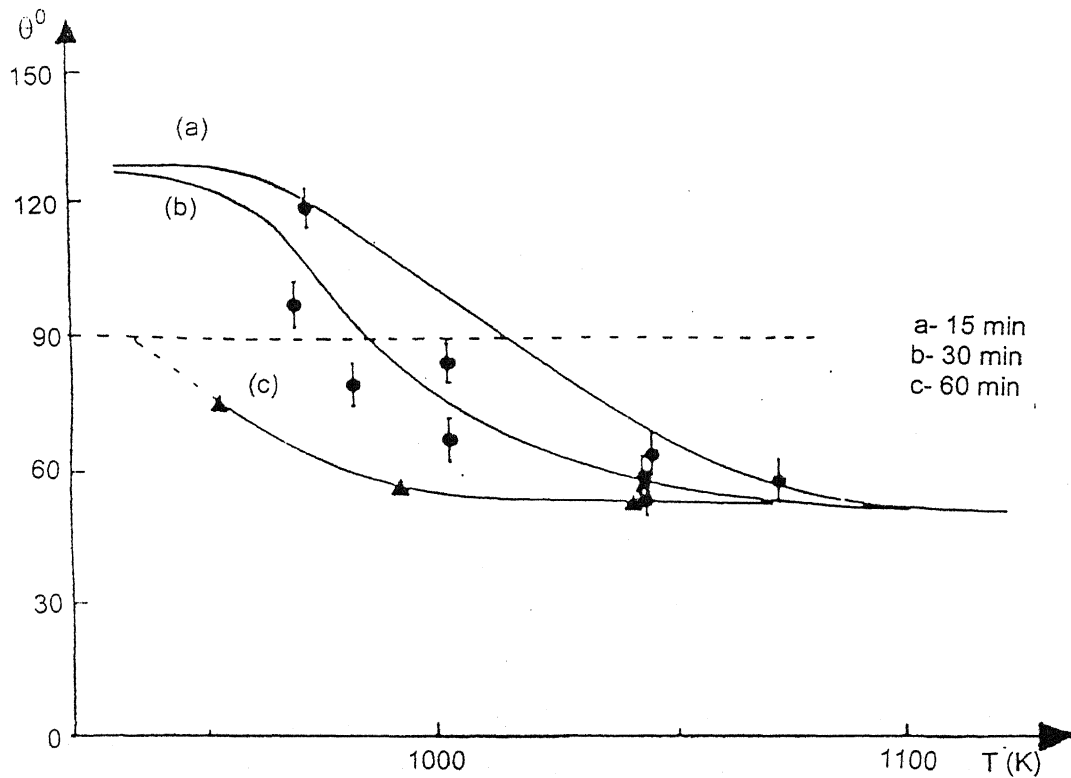
Fig. 2.3: Variation of contact angle with temperature at different time for Al/SiC system [21].

From the sessile drop experiment in a vacuum of 10^{-4} to 10^{-5} Pa, in the Al-SiC system, a "non wetting-wetting" transition was observed at a temperature that decreased as

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time increased [21]. This effect may be partially thermodynamic due to changes in surface energies and partly kinetic due to desorption and interfacial reactions [26,30].

Density of introduced particles appeared to effect the dispersion time. As the density of the particles increases, the time to dispersion increases, indicating that lift force are required and, hence, longer time is needed to lift the particle in suspension [28].

2.2.2 Interface and interface stability:

Two primary concerns guiding interface phenomena in processing are [22]:
- Preventing composite microstrutural degradation by interfacial reactions.
- Promoting interface bond formation.

For better mixing of reinforcement with liquid melt it is necessary that there should be stable interface between the matrix and the reinforcement. There are three hypothesis concerning interface between SiC and Al matrix [31].

1. There may be a SiO_2 layer at the interface.
2. There may be an Al_4C_3 film at the interface. (This may be absent in Al-Si alloy matrix).
3. Interface is formed simply between SiC and Al.

Therefore, possible interfaces in Al/Al-Si alloy – SiC particle system can be between Al and SiC, or between Al and SiO_2 surface of SiC, or between Si and SiC, or between Si and SiO_2 surface of SiC or between eutectic Al-Si and the reinforcement. In addition to these interfaces, there are interfaces within the matrix region between the constituents of matrix.

2.2.2.1 Bonding of SiC and Al:

After selection of reinforcing phase the key to controlling the properties of MMCs depends on efficient bonding between the matrix and reinforcing phase [31]. Energetic and kinetics of bond formation from the initial constituents of the composite are highly dependent on the nature of chemical interaction that takes place between the phases and can be tailored to a significant extent via compositional control of composite [22]. Also the specific crystallographic planes of the reinforcement in contact with the specific crystallographic planes of the matrix affect the bonding.

Debonding take place at the weak interface, which may be between the particle and matrix or between the matrix and other intermetallic compound. Thus it is important to select the right combination of the composite constituents to get strong interface [16].

2.2.2.2 Factors affecting interface stability: Interfacial Interactions:

- **Dissolution and reaction:** As it has mentioned in section 2.2.1.2 that mutual dissolution and reaction between two bulk phases helps in wetting. But excessive reaction may, however, lead to the consumption of particles, resulting in the weakening of the bond. Compromise must therefore be found between contradictory requirements of good wetting and absence of reaction. The best compromise between wetting and stability appears to be offered by SiC. The cause may be the presence of SiO_2 film, which seems to provide a barrier against reaction. Good wettability of SiO_2 by Al may be understood from the fact that SiO_2 is reduced by liquid Al [20].

Dissolution continues at higher temperatures and may be extensive if long duration of heating is necessary [16].

In case of Al /SiC composite, in which Mg is used to improve the wettability of particles, Mg spinel is formed as a result of the following reaction.

$$Al_2O_3 + Mg \longrightarrow MgAl_2O_3$$
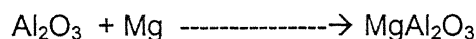
Although Mg spinel helps in wetting, its formation beyond a particular quantity has a negative impact on the interface stability. Therefore Mg should be used in controlled amount to avoid the excessive reaction. Using Al alloys can also reduce Mg spinel formation.

In case of Al/SiC composite systems, the reaction-taking place at the matrix reinforcement interface is

$$4Al + 3SiC \longrightarrow Al_4C_3$$
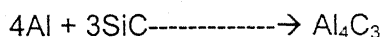
As mentioned in section 2.1.1.3 this reaction can be avoided by prior alloying the metal with Si. This can be diminished by coating of particle and also by controlling the temperature and agitation time [21,32].

- **Presence of impurity elements:** Presence of secondary alloying elements, like Fe, Mg, Sb etc. in the alloy as impurities leads to formation of intermetallic compounds such

as $FeAl_3$, Mg_2Si, etc. resulting in additional interfaces. The relative strengths and adhesion of these interfaces with matrix or with the reinforcement determines the properties of the composite [25].

2.2.2.3 Methods to improve interface stability:

♦ **Alloying addition:** Beside improvement in wetting, alloying element also promote to form strong interfaces. These interfaces improve the mechanical properties of composite [25].

♦ **Coating of reinforcing particle:** As observed in section 2.2.1.2 coating improves the interface stability by forming stable intermetallic compound at the interface. In case of Ni coating on the reinforcement and with aluminum as the matrix metal, the nickel reacts strongly with aluminum to form Ni_2Al_4. This is a very stable intermetallic compound, which stabilizes the interface. The similar behavior is exhibited in the case of copper-coated particle [16].

♦ **Atmosphere during mixing:** To prevent excessive spinel formation inert atmosphere is proposed by several workers during mixing. The atmosphere of argon, nitrogen or mixture of these gases can be used depending on the combination of the matrix and the reinforcement [16].

2.2.3 Particle recovery and particle distribution:

The studies indicate that it is difficult to obtain an absolutely uniform distribution of SiC particles in fluids by mixing. The principal factors affecting uniformity of particle distribution in casting are

1. Aggregation and skeleton formation during entry of particle in the melt due to improper wetting in a quiescent melt.
2. Setting or flocculation of individual particles and particle aggregates in the melt before and during solidification owing to density differences.
3. Localized pushing of particles by solidifying interfaces causing micro inhomogenity in particle distribution.

Beside the above factors some other points which are also responsible for particle recovery and particle distribution are:

- particle size
- percentage of particles
- rate of addition of particles
- melt degassing

2.2.3.1 Particle size and shape: Recovery of the reinforcing particles in the solidified melt depends on size of the dispersoids, as shown in Table 2.1 [27]. For the particles of finer size, there is a greater probability of agglomeration, which prevents good mixing of particles into the matrix melt. Hence for proper mixing of particles, the size distribution should be optimum [16].

2.2.3.2 Percentage of Particles: It has been observed that viscosity is a function of the reinforcement volume fraction and size. An increase in volume fraction or a decrease in size increases the viscosity of slurry. This limits the practically achievable amount of reinforcement to about 30 volume percent [15,34].

Fluidity of Al-Si alloy is higher than pure Al [7]. This property helps to incorporate more particles in the melt of Al-Si alloy.

2.2.3.3 Rate of addition of particles: The rate of addition of particles also affects the mixing of particles in the melt. If higher rates are employed, the particles do not get enough time for perfect mixing. This results in poor wetting of particles by the melt. This may also result in the agglomeration of the particles leading to low recoveries. Based on experimental investigation it has been shown that the rate of addition of the reinforcing particles into the matrix melt should be 30-40g/min for good wetting and better recovery [27].

2.2.3.4 Melt degassing: Sometimes it is necessary to degas the melt to remove dissolved gases. In case of aluminum alloys, degassing may be done by hexachloroethane or nitrogen gas prior to addition of particles. Attempts to do so after addition of particle result in complete rejection of particles. This can be attributed to the reactions between aluminum and the chlorine present in the degassifier forming compounds at higher temperature [27].

2.2.4 Solidification of particle reinforced MMCs:

Distribution of particles and mechanical properties in MMCs manufactured by casting techniques depends to a great extent on the nature of the interaction between the ceramic particles and the growing solid-liquid interface [35]. When a reinforcing particle, which is dispersed in the liquid metal encounters a growing solidification front, there are two possibilities. It may be either pushed along ahead of the advancing front, or it may adhere to the solid and be engulfed by it, remaining stationary in the solid matrix. If the particle pushing occurs, then the reinforcing phase is segregated into the last frozen, interdendritic regions of the matrix [36].

Numerous theoretical treatments on this subject have appeared in the literature over the past twenty-five years. They have established that following factors play important role in determining the critical velocity of solid-liquid interface for engulfment [37,38]:

- surface energy among particle, liquid and solid
- radius of particle
- vol. fraction of particle
- density of particle
- thermal conductivity of melt and particle
- heat diffusivity
- viscosity of melt
- concentration of solutes (dissolved impurities)

Rapid solidification of melt reduces the particle pushing phenomena during solidification. In case of thin section casting or rapid solidification, when the solid-liquid interface velocity is high, dendrite spacing will be small. Due to this, number of ceramic particles, which can be accommodated at each dendrite boundaries, will be less compared to the case where dendrite spacing is larger at slower cooling rates. It means, greater the dendrite spacing greater will be segregation due to particle pushing, results in poor distribution of particle. Therefore rapidly solidified structures give best distribution of particles. Fig. 2.4 shows particle distribution is better in case of rapidly solidified MMC as compared to conventionally cast MMCs [38,39].

A   B
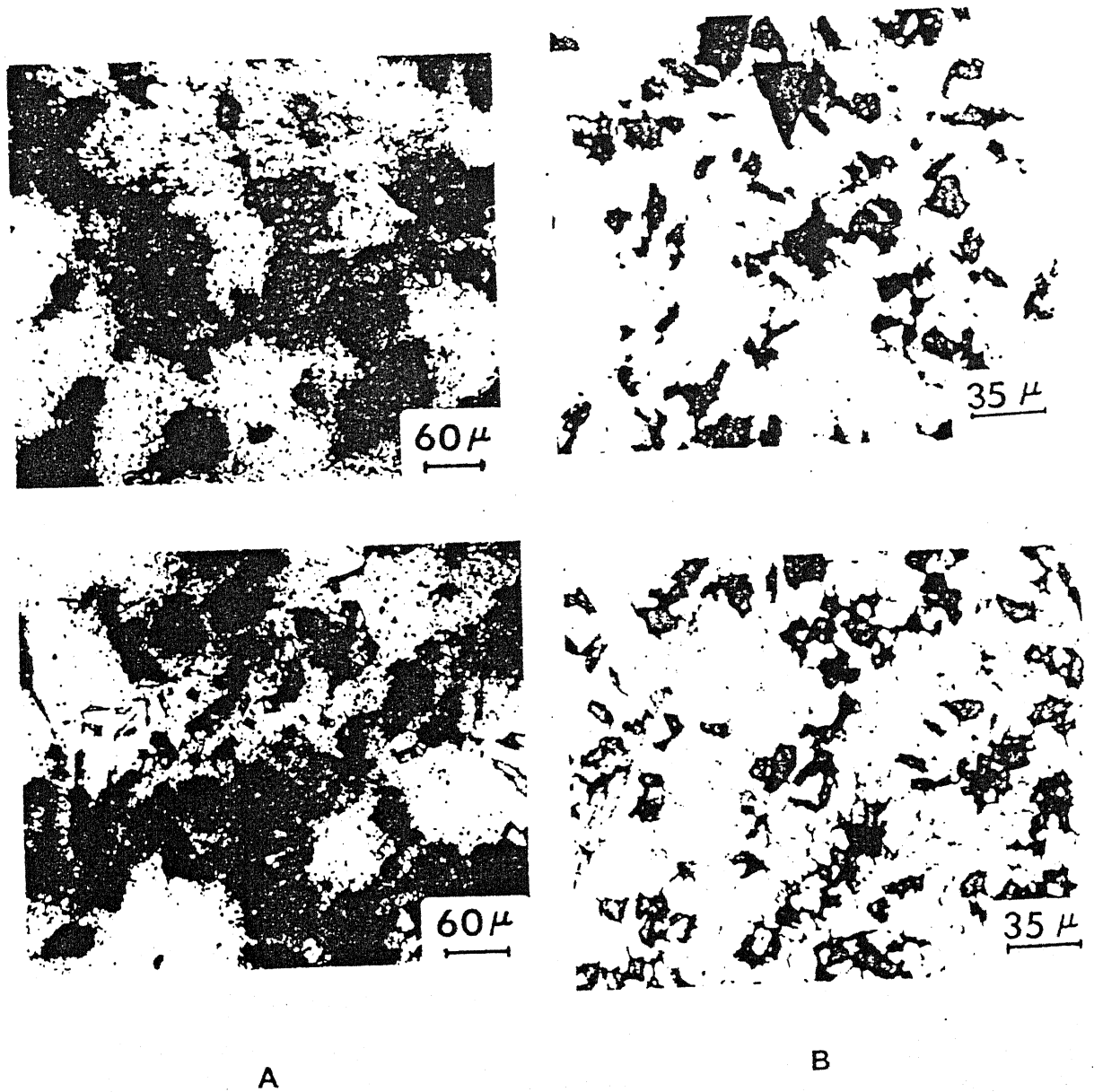
Fig. 2.4 : Photomicrographs at different cooling condition [39].
A-conventionally cast MMC, B- cast by rapid quenching

2.3 REVIEW ON STRENGTHENING AND FAILURE BEHAVIOR OF PARTICLE REINFORCED MMCs:

2.3.1 Strengthening mechanism:

The parameters controlling the mechanical properties of particle reinforced MMCs are still not understood. However, some of the factors are becoming apparent. In brief strengthening mechanisms can be envisaged as [40,41]:

1. Dispersion strengthening.
2. Elastic misfit strengthening, which arises because the elastic reinforcement cannot accommodate the plastic strain of the matrix without generating elastic stresses in the particle.
3. Coefficient of Thermal Expansion (CTE) misfit strengthening arises due to difference in CTE between the matrix and the reinforcing particles.
4. Grain size and subgrain strengthening.

It has been observed by several workers, that high dislocation density is responsible for strengthening and as mentioned above difference in CTE between matrix and particle has significant contribution in dislocation generation. However one other factor also responsible for dislocation generation, is plastic deformation during material processing.

2.3.2 Review on Tensile properties of commercially available Al-Si / SiC MMCs:

To review the mechanical properties of particle reinforced MMCs, Al based composites are briefly presented in this section. Main focus is presented on the tensile strength and yield strength of the composites.

Composite strip (2.54 mm thick) was prepared by powder metallurgy method followed by extrusion and rolling. Tensile properties of commercially available matrix alloy composites, with different volume fraction and with different kind of reinforcement were observed [42,43]. It can be seen from the Fig. 2.5 that tensile strength and yield strength of SiC/Al composites are affected by matrix alloy, reinforcement content and type of reinforcement.
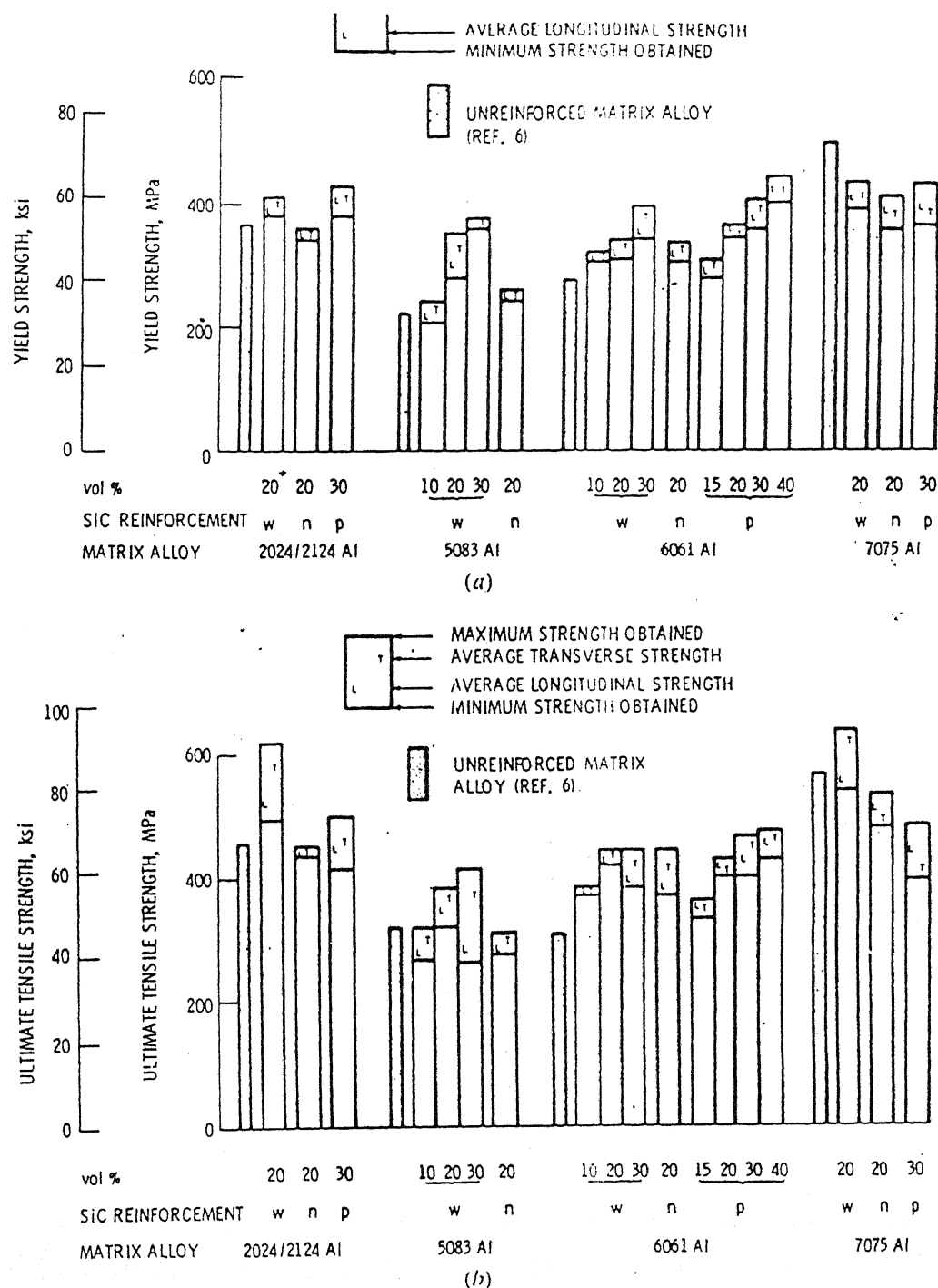
Fig. 2.5 : Effect of SiC reinforcement type and content on tensileproperties of discontinuous SiC /Al composites. (a) 0.2 pct offset yield strength, (b) Ultimate tensile strength [42].
w- whisker, n- nodule, p- particle

It was also observed that ductility of SiC/Al composites, as measured by strain to failure, is a complex interaction of parameters. However, the prime factors affecting these properties were reinforcement content, matrix alloy, type of heat treatment, and orientation [42,43].

Discontinuous SiC/Al composites continued to show an advantage over conventional aluminum alloys at elevated temperatures. It was observed that, at temperatures of less than 240°C, the composites failed with relatively little plastic flow and no necking. Above this temperature composite showed the abrupt change in behavior and exhibited significant necking and failed at plastic strains of about 10 pct. The above investigation was carried out for short time exposure of 10 minutes. It has also been observed that SiC/Al composites can probably be used effectively for short time exposures up to 240°C and for long time exposures up to 204°C [42].

2.3.3 Failure mechanism of particle reinforced MMCs:

A major limitation of composites is their limited tensile ductility; and so a considerable amount of research effort is being expanded on fracture. Ductile fracture of the conventional alloys is considered in terms of microvoid coalescence model (MVC). It has been observed that void nucleation in unreinforced alloys occur at constituent particles, either through particle failure, or through interface decohesion, which grow by coalescence of voids.

Failure mechanisms influencing the constitutive response and fracture resistance of the MMCs can be broadly classified into three groups [43-48]:
1.   Brittle failure of the reinforcement by particle fracture.
2.   Debonding and fracture along the interface between the matrix and the reinforcement.
3.   Ductile failure by nucleation, growth and coalescence of voids in the matrix.

The degree to which these failure mechanisms individually and collectively influence the overall deformation and fracture resistance is strongly dictated by factors as diverse as:
•   The size, shape, concentration and spatial distribution of the reinforcement.
•   The concentration of impurities presents in the constituent phases of the composites.
•   The processing and heat treatment procedures, to which composite is subjected prior to mechanical properties evaluation.

- The thermal and chemical environment in which mechanical properties are evaluated.

- The coating, if applied to the reinforcement with the specific objective of modifying the interfacial characteristics.

Flame and Arsenault [43] studied the fracture behavior of SiC/Al composite and concluded that, it has features of both brittle (limited crack blunting, microcracking in front of the crack tip, confinement of the fracture process to a very narrow band) and ductile (dimple morphology of the fracture surface) mechanisms. Also fracture process is matrix controlled upto SiC particle sizes of 20 μm and above, fracture of SiC begins to dominate.

Both microscopic (short range) and macroscopic (large range) residual stresses are known to influence the mechanism. Thermal conductivity mismatch in composite result in the average stresses, which are tensile in the matrix and compressive in the reinforcement. These stresses can have a pronounced effect on the local crack path. Macroscopic residual stresses, such as those caused by quenching in the platform tend to have long range effects, for example causing the crack front to bow [44].

It has been observed that triaxial stresses are responsible for both void nucleation and void growth mechanism. The deformation response of the particle-clustered region was found different from the rest of the composite, because within the clusters the far field applied stresses are no longer controlling. The elastic particles constrain the deformation of the matrix adjacent to them, and this resulted in complex triaxial stresses being exerted on the matrix within the clusters. Thus critical factor for tensile failure is matrix voiding between closely spaced particles, and in the matrix within particle cluster.

CHAPTER 3

Experimental procedure

In this chapter, the experimental procedure employed in this investigation is described. First, the preparation of the composite melt is dealt with. This is followed by technological problems experienced during composite preparation and remedial measures employed. Brief description on continuous strip casting method by Single roll strip caster and evaluation of structure and mechanical properties of these strips are discussed at the end.

Composition of the matrix used in composite preparation is given in Table 3.1. SiC particles (<25 μm, 44-53μm and 53-75μm average diameter), with different weight percentage were used.

3.1 COMPOSITE MELT PREPARATION:

Following the principle of Vortex method, experiments were carried out for preparation of composite melt. Schematic diagram of experimental set-up is shown in the Fig 3.1. It consists of resistance furnace, which is heated by SiC rods. Crucible is used for melting. Melt is stirred through a stirrer from the top with the help of motor.

Steps involved in the melt preparation are:

- melting of the aluminum alloy
- addition of degassifier
- addition of Magnesium
- preheating of particles
- incorporation of particles in the melt
- stirring of the melt

Table 3.1 : Composition of the matrix used in the experiments:

Element	Al	Si	Cu	Mg	Fe
Wt. %	84.46	15	0.22	0.09	0.23

Stirrer Motor

Furnace

Heating Filament

Furnace
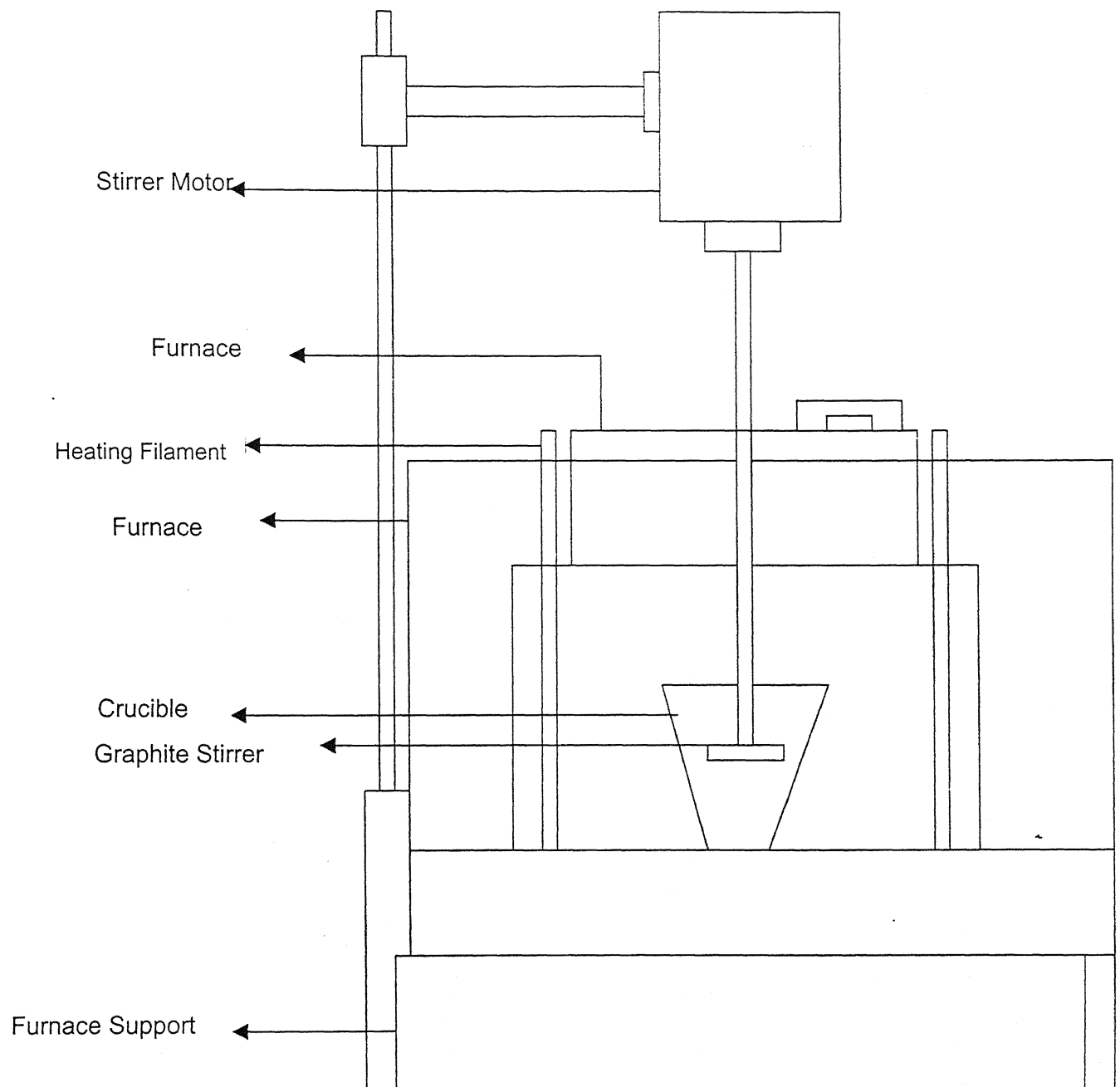
Crucible

Graphite Stirrer

Furnace Support

Fig. 3.1 : Schematic diagram of experimental set-up

3.1.1 Melting of the aluminum alloy: First the charge was heated above its

melting point. Care was taken to minimize the time required to attain the required temperature keeping in view the high oxidizing characteristics of aluminum alloy. The charge was melted in a resistance-heating furnace shown in Fig. 3.1. When the temperature reaches about 740°C, slag and dross, if any, was removed.

3.1.2 Addition of degassifier: After complete melting, liquid melt was degassified

with the help of hexachloroethane to remove the dissolved gases.

3.1.3 Addition of Magnesium: A specified quantity of magnesium (0.5~1%) was

added to improve the wetting of particle with the melt. Initially magnesium was added in the form of turnings, wrapped in an aluminum pouch, which is tied on to one end of the graphite rod with a thin aluminum wire. The graphite rod was then inserted into the melt, which was manually stirred with the same rod for about 5 minutes. There were a few problems encountered in this step, however, these could be resolved by using magnesium granules in place of turnings; this is discussed in detail in the section 3.2. Care was taken to minimize the heat losses during this whole process. After some time, any slag, if present, was removed.

3.1.4 Preheating of reinforcement particles: The preheating removes

the volatile contaminants present, if any, on the surface of the particles. This aspect has been dealt with in detail in the literature review. Particulates were heated at 900°C for one hour in a resistance-heating furnace. The heating rates of both furnaces, i.e. the furnace used for melting alloy and the one used for preheating the particles were synchronized such that the required temperatures were reached almost simultaneously in both the furnaces. After one hour the ceramic particles were removed from the furnace and incorporated into the melt.

3.1.5 Incorporation of particles in the melt: This was perhaps the most

important step in the preparation of the melt for composites. The time lag between removal of the ceramic particles from the preheating furnace and addition into the melt was kept to a minimum to avoid excessive cooling of the particles. The particles were

added into the melt while the melt was being continuously stirred with the graphite stirrer from the top. Care was taken to ensure that the particulates were added into the vortex formed by the stirring action for better mixing. The rate of addition was maintained at 30-40 g/min for better recovery. The temperature of the melt was maintained between 740^0C-760^0C.

3.1.6 Stirring: For the stirring purpose, a stirrer motor of 1 hp was used. The speed

of the stirrer was kept between 400-450 RPM. Rotational speed touched the superior limit during particle incorporation. Before starting the stirring process, the blades and the stirrer shaft were preheated in the furnace to avoid thermal shocks and the consequent reduction in the melt temperature. After addition of the reinforcing particles, stirring was continued for 15-20 minutes.

Temperature was continuously monitored during the whole process of the melt preparation using a thermocouple. The slag and other impurities floating onto the melt were removed. Temperature of the melt was measured before transferring it in to the tundish of the caster.

3.2 TECHNOLOGICAL PROBLEMS EXPERIENCED DURING COMPOSITE MELT PREPARATION AND REMEDIAL MEASURES EMPLOYED:

While preparing melt by vortex method a number of problems were encountered, such as poor dispersoid-matrix bonding, inhomogeneous distribution of particles, aggregation and skeleton formation after particle addition, etc. As a result, mechanical and tribological properties of the composites deteriorated significantly and sometimes addition of dispersoid phase produced a negative effect [49].

These problems arose primarily because of the fact that the reinforcement being ceramic material has a poor wettability with molten metal/alloy. As observed in chapter 2, the type of the dispersoid phase and the nature of interface formed between the matrix

and the reinforcement are of great importance to effect load transfer and crack resistance of the MMCs during deformation. So, care must be taken in the melt preparation to get strong interface.

It is to be noted that a reinforcement particle has an envelop of air /gas, which, if not broken, prevents interaction of the melt with the dispersoid causing interfacial porosity. The dispersoid surrounded by the gaseous envelope has reduced apparent density. This causes floatation of the dispersoid phase to the top of the melt. In such situation segregation / coagulation and inhomogenous distribution of the dispersoid phase in the matrix are experienced. These ultimately impair the properties and quality of the composite. Entrapped gases also cause the generation of porosity. Hence degassing of the melt must be done to remove the entrapped gases [50].

With the help of the vortex technique, partial breaking of the air/gas envelope occurs, which improves the intermixing and wetting tendency of the dispersed particles with the matrix. During experiments a few other modifications were introduced, which reduced the above problems. These modifications included:

♦ use of angular blade teeth in the stirrer blade to create proper vortex instead of using flat teeth blade,

♦ making the preheated particles freely flowing by spreading them, (to avoid agglomeration of particles) before dispersion,

♦ checking of proper vortex during particle addition by adjusting the stirrer speed,

♦ using magnesium in the form of solid granules instead of Mg turnings to reduce the burning loss of Mg.

3.3 CASTING OF COMPOSITE STRIP:

The design and fabrication of Single roll continuous strip caster developed by Mehrotra and co–workers; and employed in this investigation, are presented in detail elsewhere [51,53]. A brief description of the caster and its operation are presented in this section.

3.3.1 : Caster drum and its accessories :

A Schematic representation of caster assembly is shown in Fig. 3.2. The main components of caster include:

♦ tundish / reservoir
♦ caster drum assembly
♦ water spray system
♦ knife edge
♦ stepper motor
♦ microprocessor based control system
♦ thermocouple assembly

3.3.1.1 Tundish/ Reservoir :

Tundish, which is made up of a fire clay brick, primarily acts as reservoir to hold the molten metal, and feeds it on to the rotating drum in a controlled manner. The rate of flow of the metal to the drum, which is determined by the rate at which the metal is removed in the form of the solidifying strip, is controlled by controlling a constant metal head in the tundish. It has rectangular opening at the bottom that acts as an outlet for the molten metal. The tundish is placed very close to the caster drum without touching it to avoid scratching of the drum surface. At the same time it is ensured that gap between the drum surface and the tundish is not large to lead to the leakage of the molten metal through it.

3.3.1.2 Caster drum assembly:

The caster drum is made of high purity (99.99%) copper. It is hollow cylinder with both ends open. It is cooled internally by water spray. The total length is divided in two portions. One is the caster drum portion and the other one is water outlet portion, which is drilled with holes on its surface and provides an outlet for cooling water. One end of the caster drum is connected to the shaft of the microprocessor controlled stepper motor, which can vary the rotational speed of the caster drum. The other end of the drum is connected to a water pipe line and also holds the water spray assembly inside the drum. The caster drum and water outlet portion of the drum are sealed on the outer surface by a brass ring so that the exit water does not come in contact with the molten metal or the solidifying strip at any time.

3.3.1.3 Water spray assembly:

Water spray assembly consists of four nozzles placed at right angles to each other and in two rows. The water spray covers almost the entire surface area of the caster drum to ensure its uniform cooling. The spray nozzles are specially designed such that each nozzle generates a fully developed water cone with cone angle of about 70^0. The nozzles are fitted through the manifolds on the horizontal stainless steel pipe, which passes through the brass flange on one of the openings of the caster drum. This pipe remains stationary when the drum is rotating.

3.3.1.4 Knife edge :

The Knife edge, made of aluminum sheet, is fixed on to the platform on which the cast sheet moves after solidification. The main function of knife edge is to peel off the solidified sheet from the caster drum. The position of the knife edge can be adjusted through its mount by giving it required horizontal and vertical movements.

3.3.1.5 Stepper Motor :

The motor used to rotate the drum to any specified rotational speed is a Uni-step motor. It runs with 12 volt D.C. supply. The motor can be directly coupled with caster drum through a shaft or it can be coupled through a gear assembly to obtain wider range of the speed of rotation.

3.3.1.6 Microprocessor based control system :

The microprocesor kit, having total memory capacity of 64 K Bytes is used to control the stepper motor. The programme to control the speed of the stepper motor through microprocessor has been indigenously developed.

3.3.1.7 Thermocouple assembly :

Provision is made to continuously measure the temperature of the caster drum wall at two points during the casting. These temperatures are measured using chromel-alumel thermocouples. One of these is at the center of the drum wall at the mid point along the length of the casting zone while the second location is almost at the water cooled surface of the drum.
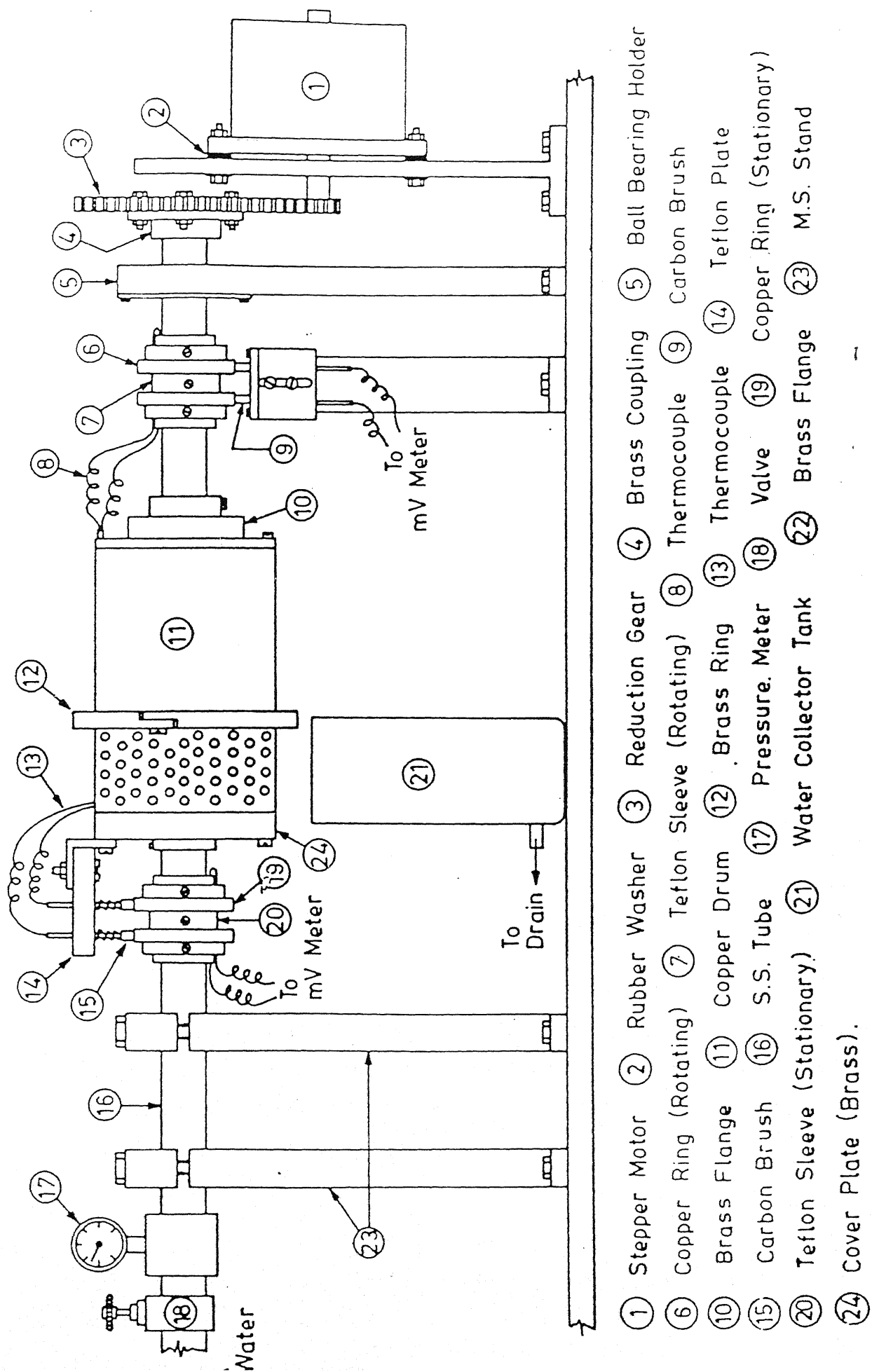
Fig. 3.2: Schematic diagram of Single roll continuous strip caster Assembly

1. Stepper Motor
2. Rubber Washer
3. Reduction Gear
4. Brass Coupling
5. Ball Bearing Holder
6. Copper Ring (Rotating)
7. Teflon Sleeve (Rotating)
8. Thermocouple
9. Carbon Brush
10. Brass Flange
11. Copper Drum
12. Brass Ring
13. Brass Ring
14. Thermocouple
15. Carbon Brush
16. S.S. Tube
17. Pressure. Meter
18. Valve
19. Copper Ring (Stationary)
20. Teflon Sleeve (Stationary)
21. Water Collector Tank
22. Brass Flange
23. M.S. Stand
24. Cover Plate (Brass).

3.3.2 Strip production by Single roll continuous strip caster:

To start the casting operation caster is switched on at a prespecified speed of rotation using the stepper motor. A prespecified water flow is set for cooling purpose. Melt is poured into the preheated tundish to minimize the thermal shock. Pouring is done in such a way that the melt level in the tundish reaches the marked level and remains constant during the entire casting process. Melt pool is formed in the annular space between the rotating caster drum and the tundish wall. As soon as the drum comes in contact with the melt, formation of solid strip commences at the drum surface. The strip is gently withdrawn from the pool and brought over the drum surface with the help of dummy bar, which is a bent aluminum strip of the same width as that of the strip. The strip is peeled off from the drum with a knife-edge when it reaches the top of the drum. It then travels on its own to the guiding platform. The casting process continues as long as the melt is poured from the crucible. The strip is then allowed to cool down in the ambient conditions. The operating parameters such as speed of rotation of caster drum, water flow rate are noted and marked on the strip.

Schematic diagram of caster drum operation is shown in Fig. 3.3.

3.4 OPTIMIZATION OF PROCESS PARAMETERS FOR EXPERIMENTAL INVESTIGATION:

A number of problems are associated with the liquid metallurgy route of composite strip production (vortex dispersion method of particle reinforced melt preparation combined with SRCSC strip production method), consequently attempts to improve the process need to include optimization in melt preparation as well as in SRCSC method of strip production.

As it became clear from the literature review that many of these can be controlled only at the cost of the other, and it became important to adjust the parameters such that they did not impair the required properties.
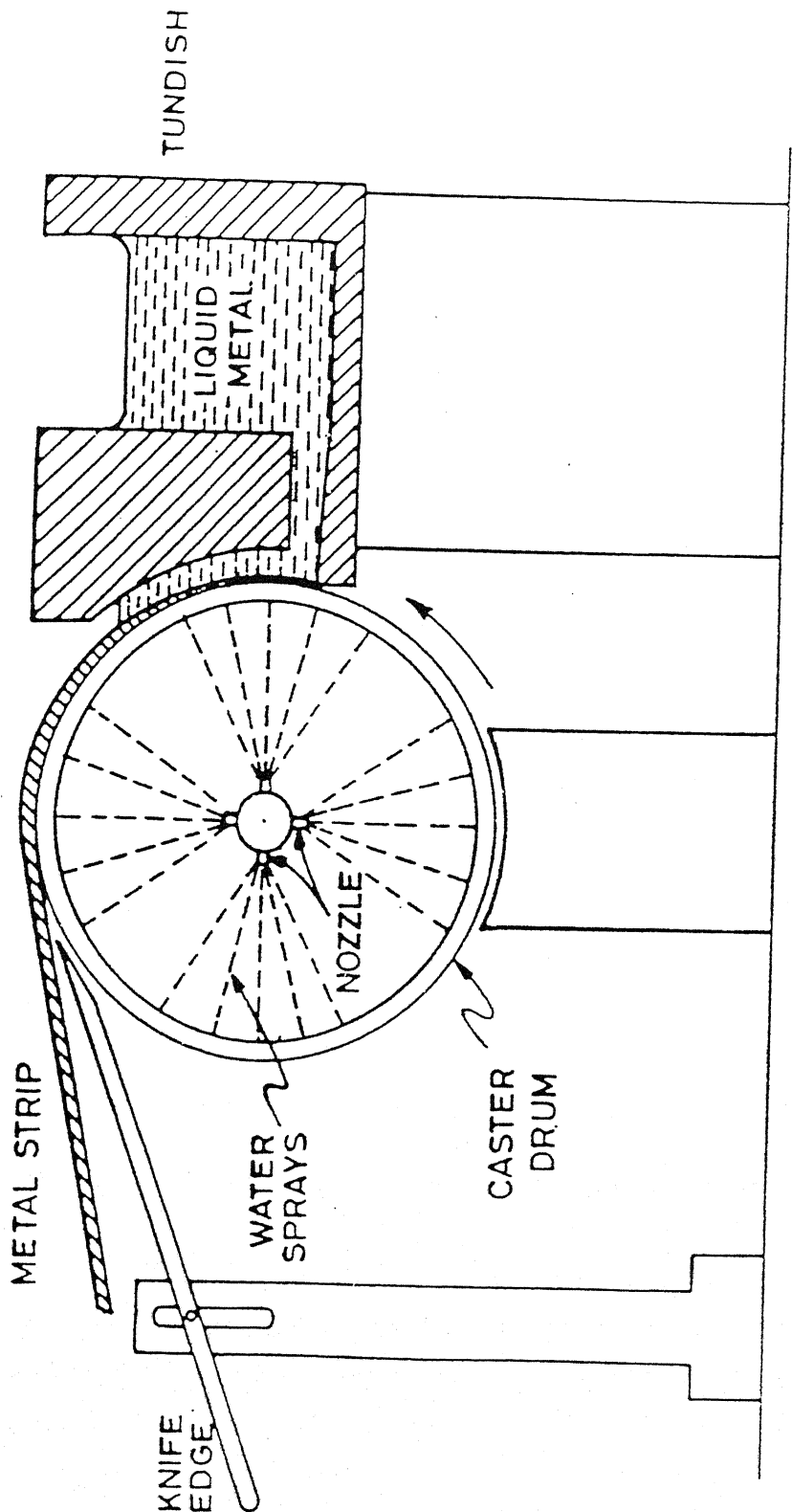
Fig. 3.3: Schematic diagram of caster drum.

3.4.1 Melt preparation: Several operating parameters need to be optimized during melt preparation to achieve the aim of good wetting, high bond strength, homogeneous distribution of particles in the melt, etc. It needs to avoid gas entrapment, presence of inclusions and segregation of particles.

In composite melt preparation part of the experiments, variables which could be optimized were broadly divided into two groups:

1. Process based parameters: These included temperature of the melt, melting time, rpm of the stirrer, duration of stirring, and pouring temperature.
2. Reinforcing particle based parameters: These included particle size and amount of particulates to be added.

Several experiments were performed by changing the parameters intelligently in a manner so as to reduce the detrimental effects on properties of the composite. First Process based parameters were optimized. After optimization of process based parameters, these were selected to study the effect of reinforcing particle based parameters. Process based parameters were optimized on the basis of microscopic evaluation such as distribution of particles, homogenity, recovery of particles, etc.

3.4.2 Strip casting : Several trials, has been made on the strip caster to optimize the caster parameters such as height of the liquid melt in the tundish, tundish nozzle gap, cooling water flow rate, etc. After optimization of these parameters, effect of speed of rotation of caster drum on the properties of the strip were studied.
Experimental parameters are given in Table 3.2.

3.5 EVALUATION OF COMPOSITE STRIPS:

One of the main features of this investigation has been the evaluation of the microstructure and mechanical properties of the composite strips produced by the single roll continuous strip caster. In microstructure evaluation, distribution of reinforcing particles and observation of strip quality were performed. Mechanical properties examined included tensile strength, yield strength, ductility both at ambient and elevated temperature and hardness.

Table 3.2 : Experimental parameters :

Process steps	Variables	Range
Reinforcement particle Variables	Particle size	<25-75μm
	Quantity of particle	5-12 %
Particle incorporation Variables	Preheating of particle	900⁰C for 1 hr
	Temperature range during addition of particle	740-780⁰C
	Rate of particle addition	30-40 gm/min
Mg addition	Quantity of Mg	0.5 – 1 %
	Temperature of Mg addition	720-740⁰C
Stirring parameters	Rpm of stirrer	400-450
	Duration of stirring	10-15 min
Casting parameters	Pouring temperature	640⁰C
	Height of liquid metal the tundish	10-40 mm
	Tundish nozzle gap	10-20mm
	Cooling water flow rate	0.4-0.8 GPM
	Speed of rotation Caster drum	12-26 rpm

3.5.1 Microstructure evaluation:

3.5.1.1 Sample Preparation: To observe the distribution of the reinforcing particles in the matrix, microscopic examination of specimens was carried out. Four to five samples were cut from each strip from different positions so as to see whether or not the distribution of particle is uniform throughout the strip.

For microscopic examination samples were mounted using cold setting compound. The usual grinding and emery polishing was done with paper of grade 1/0 to 4/0. This was

followed by wheel polishing, first with 1μ and finally with 0.3μ alumina suspension. The wheel polishing was continued until a mirror surface is obtained. These samples were then employed for microscopic examination. Samples for electron microscope were prepared similarly, the only difference is that graphite coating on sides of the specimen was given to make the specimen conductive with mount.

3.5.1.2 Optical Microscopy: Under optical microscope, distribution of the particles was observed and recovery of particles was calculated by quantitative metallography.

3.5.1.3 Electron Microscopy: Electron Probe Micro Analyzer (JEOL JXA 8600MX) and Scanning Electron Microscope (SEM) (JSM 840A) were used to observe the distribution of particles, shape of the particles, and checking of agglomeration, inclusions, if any. The composition analysis was also carried out at various locations in the matrix. Photomicrographs were taken at various magnifications to reveal the distribution of particles.

3.5.2 Mechanical properties Measurements:

For these evaluation samples were cut to the required dimensions from the strips in the longitudinal as well as transverse direction. It was ensured that these samples were flat, without any protrusions and cracks on the surface. As the shearing normally results in some changes in the mechanical properties, the specimens were cut with a power saw. Standard dimensions of the specimen for room temperature and elevated temperature is shown in Fig. 3.4 and Fig. 3.5 respectively.
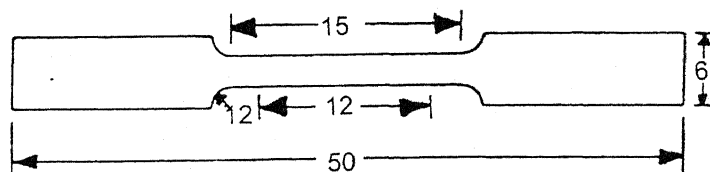
Fig.3.4: Standard tensile test specimen [16].   THICKNESS OF SPECIMEN -2 mm
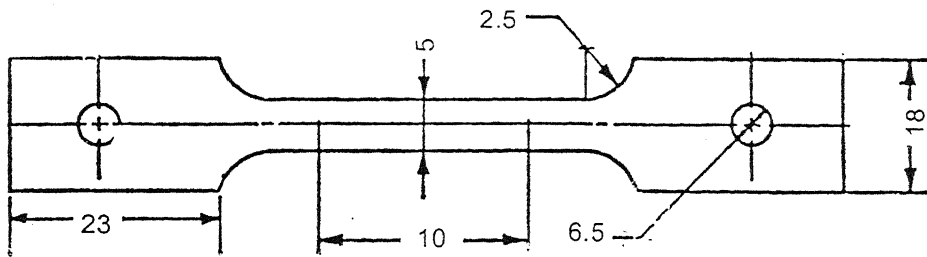(all dimensions are in mm).

Fig.3.5: Standard tensile specimen for elevated temperature tensile test [52].
(all dimensions are in mm).

3.5.2.1 Strength and Ductility: Tensile testing was carried out on the INSTRON 1195 tensile testing machine. In these tests automated load verses displacements plots were recorded and the ultimate tensile strength, 0.2% offset yield strength and percentage elongation were calculated. For elevated temperature tensile test all the tensile properties were calculated similarly. Only difference in the testing process was that a heating furnace is attached onto the same testing machine.

3.5.2.2 Hardness: Hardness was measured on Rockwell Hardness Tester. Well-polished strip sections were taken for hardness measurements.

CHAPTER 4

Results and Discussion

After optimization of process parameters, several experiments were performed to observe the effect of particle size, percentage of particles and speed of rotation of the caster drum on the microstructure and properties of composite strips. Results of these experiments are discussed in this chapter.

4.1 RECOVERY AND DISTRIBUTION OF PARTICLES:

Recovery of particle was calculated on the basis of quantitative metallography. The percentage recovery at different percentage of reinforcing particulate and particles of different sizes are shown in Fig. 4.1. Higher recovery is seen for coarser particles as compared to that for the finer particles. Better wetting and dispersion are responsible for higher recovery of coarse particles. Tendency to form agglomerates and increased viscosity of the melt are the likely reasons for lower recovery in case of fine particles [27].

Particle distribution was qualitatively observed by metallographic examination, which showed that the distribution of particles is homogeneous for the coarse particles (53-75μm). Some agglomeration of particles has been observed in the case of smaller particles, which can be seen, from micrographs shown in Fig.4.2. Effect of rotation of caster drum on particle distribution was also observed. Better distribution of particulate was found, at higher rpm (20 rpm) than at lower rpm of caster (12 rpm), which can be seen from the micrographs shown in Figs. 4.2 and 4.3.

4.2 SURFACE QUALITY:

From the visual examination it has been observed that the roll side surface was smooth while the topside surface was comparatively rough. This is in agreement with the study carried by Rao on aluminum strips [53].

Thickness of the strip affects the surface quality of the strip. As shown in Table 4.1 thickness of the strip increases with decrease in rpm of caster drum, which resulted in

drum. Thicker strip sections offer more resistance to the heat transfer resulting in reduced solidification rate, which provides time for light impurity atoms to float to the top. The stirring of the liquid metal during the incorporation of the reinforcing particles also contributes to the roughness of the strip. The stirring of the liquid metal entraps the atmospheric gases in the melt, which may try to escape during the solidification process leading to a rough strip surface.

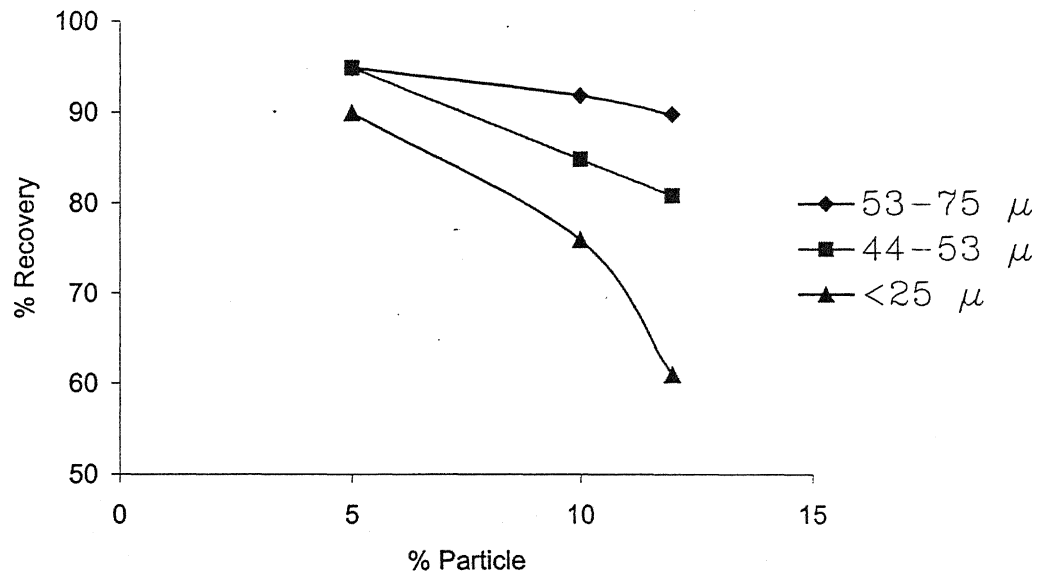
Fig. 4.1: Recovery of particle added to the melt.

Table 4.1 Thickness of strip at different rpm of caster drum :

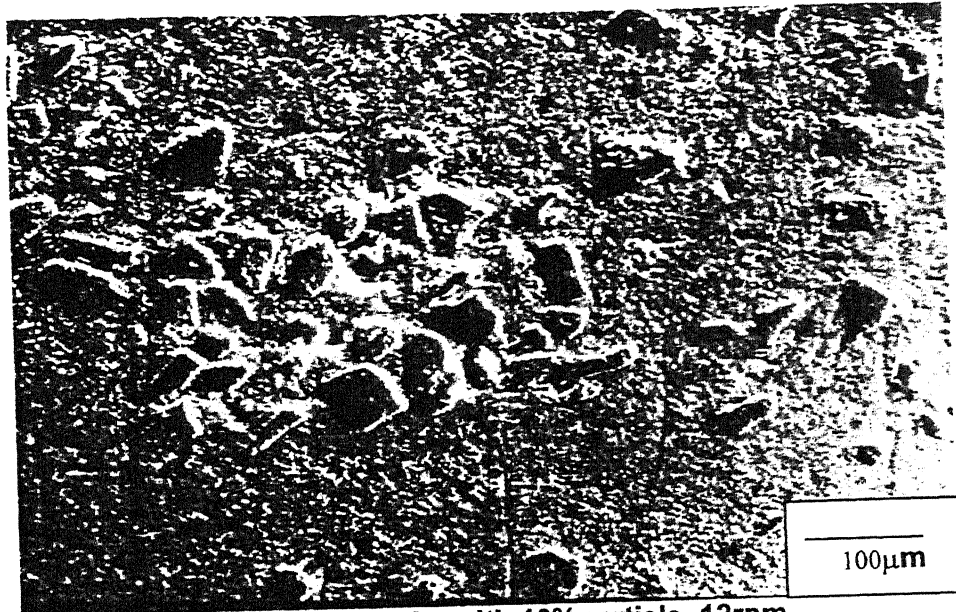| RPM of caster drum	Thickness (mm)
32	1.6
24	2.2
20	2.8
16	3.3
14	3.8
12	4.2

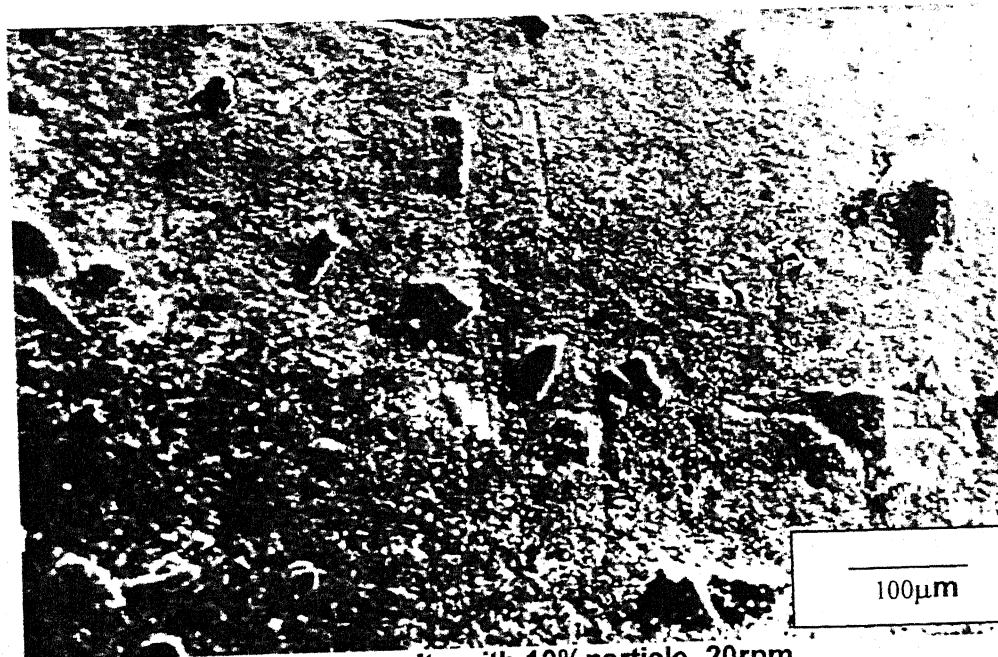Fig. 4.2 : Al-Si /SiC$_p$ composite with 10%particle, 12rpm.

Fig. 4.3 : Al-Si /SiC$_p$ composite with 10%particle, 20rpm.

4.3 MICROSTRUCTURE AND INTERNAL QUALITY OF COMPOSITE STRIPS:

Beside the reinforcing particle distribution; porosity, segregation, inclusions are the other important factors, which affect the mechanical properties of the produced strips. Preventive measures must be adopted to avoid the detrimental effects of these parameters. The settling problem that generally occurs in gravity casting processes was avoided in single roll continuous strip casting method. Optimizing the various factors, the following results were obtained.

4.3.1. Effect of particle size:

The microstructural study of particle distribution in different sections of the strip has shown that the segregation was more pronounced when small particle size were used. Distribution of particle was found to be uniform with coarse particles as shown in Figs.4.4, 4.5 and 4.6. Segregation was observed in case of small particle reinforcement, as is evident in micrographs shown in Fig.4.2 and Fig. 4.7.

4.3.2 RPM of stirrer:

Two important effects of the rpm of the stirrer were observed. At lower rpm particles did not go into the melt and floated to the top of the melt. This impairs particle recovery. At higher rpm the amount of dissolved gases increased due to high vortexing. These two reverse factors were qualitatively considered and based on visual observations the rpm of the stirrer was optimized.

4.3.3 Temperature of melt and stirring time: It has been observed that high temperature and long stirring time resulted in erosion of crucible, stirrer blade and rod, which increases the inclusion content in the strip. This is enhanced by high temperature. Inclusion content was observed in case of long stirring time (30 min). Stirring time was reduced to 5-10 min, which was found to be insufficient to incorporate the particles completely in the melt. Stirring time of 15-20 min, combined with 740-760^{0}C temperature of furnace was found optimum.

4.3.4 Effect of rpm of caster drum: The segregation level in the strip was

observed to decrease due to an increase in the rpm of the caster drum, resulting in good distribution of particle. It can be seen from Figs. 4.7 and 4.8, that at 16 rpm segregation is less compared to the microstructure shown in Fig.4.2 for 12 rpm. Distribution of particle was observed good at rpm 20 of the drum caster, which can be seen from Figs. 4.3 and 4.9.

RPM of the drum also affects the internal porosity. It was observed that the strip produced at lower rpm has qualitatively lesser porosity as compared to the strip produced at higher rpm. This can be attributed to higher rate of solidification at higher speeds that allows lesser time for the entrapped gases to escape.

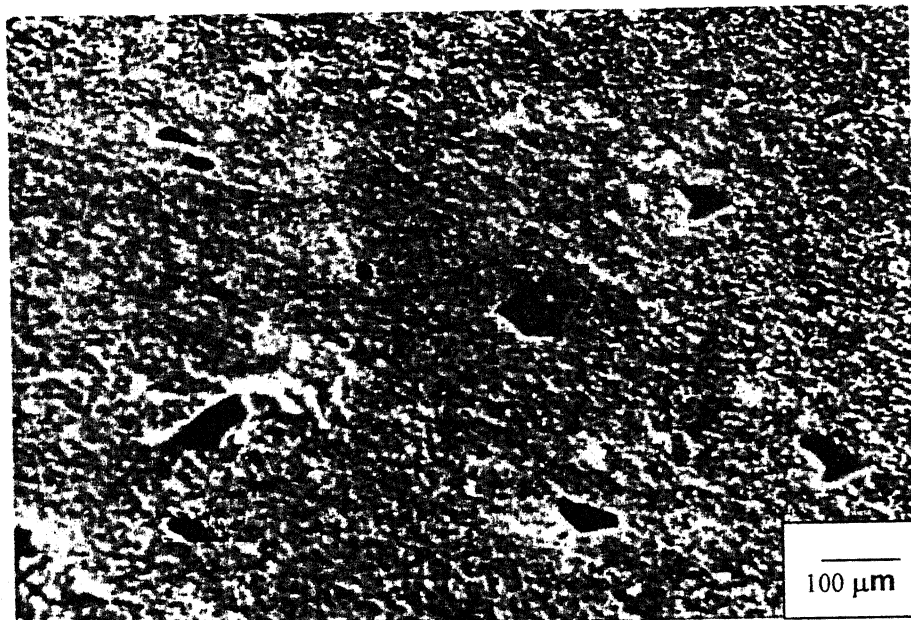Fig. 4.4 : Al-Si /SiC$_p$ composite with 10%particle, 16rpm.

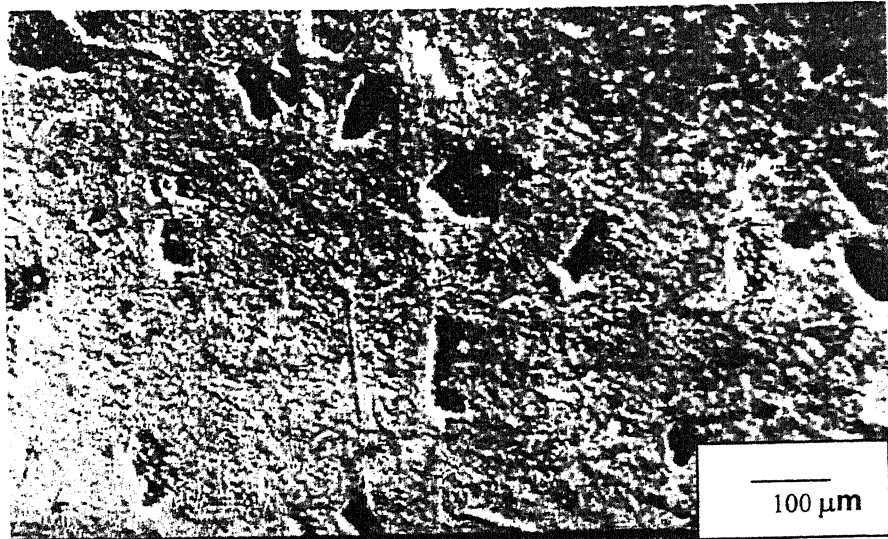Fig.4.5: Al-Si /SiC$_p$ composite with 5%particle, 16rpm.

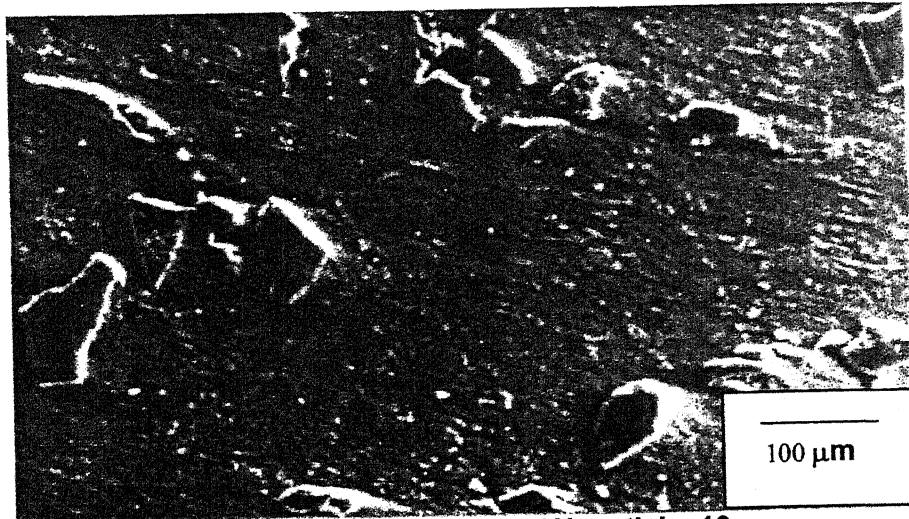Fig. 4.6 : Al-Si /SiC$_p$ composite with 10%particle, 16 rpm.

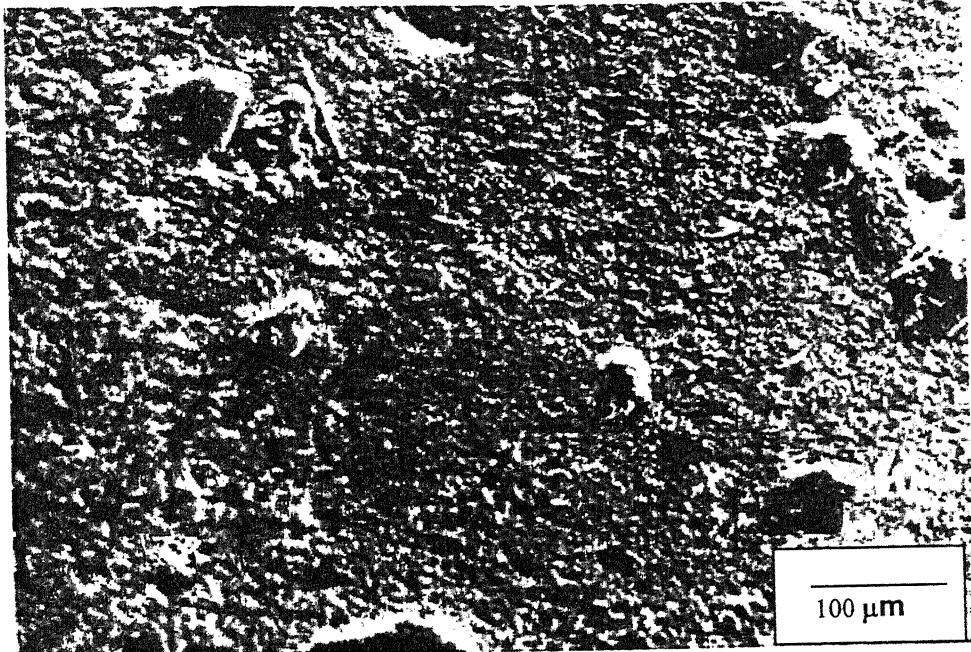Fig. 4.7: Al-Si /SiC$_p$ composite with 10%particle, 16rpm.

Fig. 4.8 : Al-Si /SiC$_p$ composite with 10%particle,16rpm.

Fig. 4.9 : Al-Si /SiC$_p$ composite with 10%particle, 20 rpm.

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4.4 MECHANICAL PROPERTIES:

The tensile properties and hardness of the strips produced at various caster drum speeds, and for various wt. percentages of particles in the composite were measured. To observe the high temperature strength of the composite strip tensile test were performed at elevated temperatures (150 °C, 200 °C, 250 °C and 300 °C). The results of these tests are discussed below.

4.4.1 Effect of reinforcement:

The mechanical properties of composite strips are affected by the size of particles and percentage of particles added to the melt. The effects of particle size and weight percentage of particle on the longitudinal and transverse tensile strength are shown in Figs.4.10 and 4.11 respectively. Similarly the effect of particle size and weight percentage of particle on the longitudinal and transverse yield strengths are shown in Figs.4.12 and Fig.4.13 respectively. Elongation of composite strips produced using particles of different sizes and with different weight percentages are shown in Fig.4.14.

Particle size has a significant effect on strength of the composite. It can be deduced from the figures that the strength of the sheet is higher in presence of smaller particles. This is essentially due to improved bonding of particles with the matrix, which prevents premature failure of composites. The fracture of particles is also avoided when the particle size is smaller in size. In case of large size particle reinforcement, premature failure was observed. This may either be due to brittle failure of particles or by debonding and fracture along the interface between the matrix and the particle. Debonding is favored for large size particles due to the presence of microcracks [43,44].

For good strengthening < 25μm ceramic particles are suited best. There was no significant contribution to strengthening when 44-53μm size particles were used. The strength of composite is seen to be decreasing due to premature failure, when particle of size >53 μm with high percentage were employed. In case of yield strength, a little improvement in strength can be seen with 44-53μm size of the particle. This is due to particle size factor, which is less predominant at lower loads and with smaller size of particle compared to coarse particles (>53μm).

Fig. 4.10 : Schematic diagram of tensile strength (L) at different
wt. fraction of particles (rpm of drum caster-16)
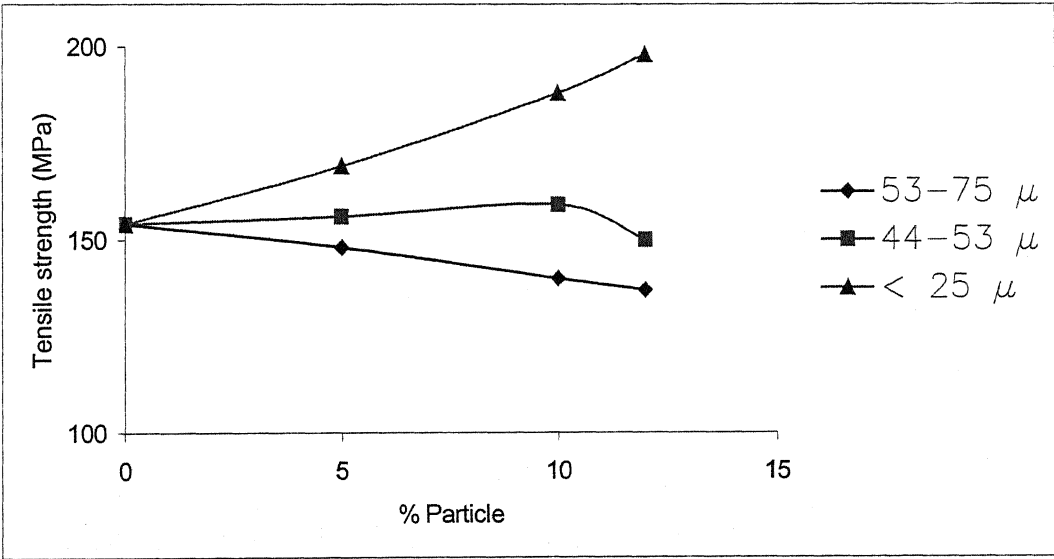
Fig. 4.11 : Schematic diagram of tensile strength (T) at different
wt. fraction of particles (rpm of drum caster-16)

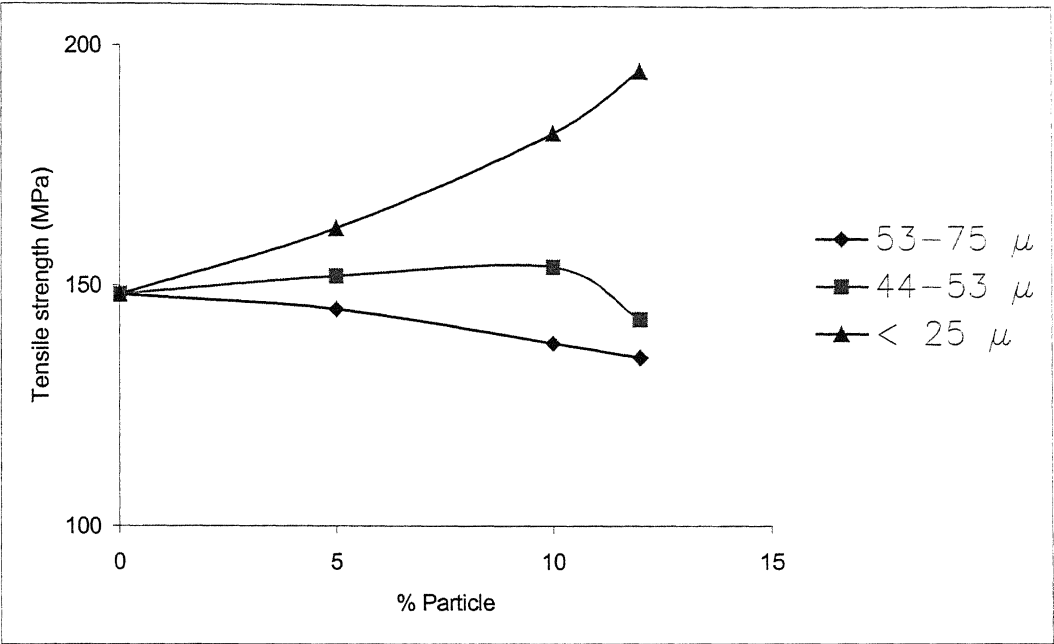[L : Longitudinal direction, T : Transverse direction]

Fig.4.12 : **Schematic diagram of yield strength (L) at different**
wt. fraction of particles (rpm of drum caster-16)

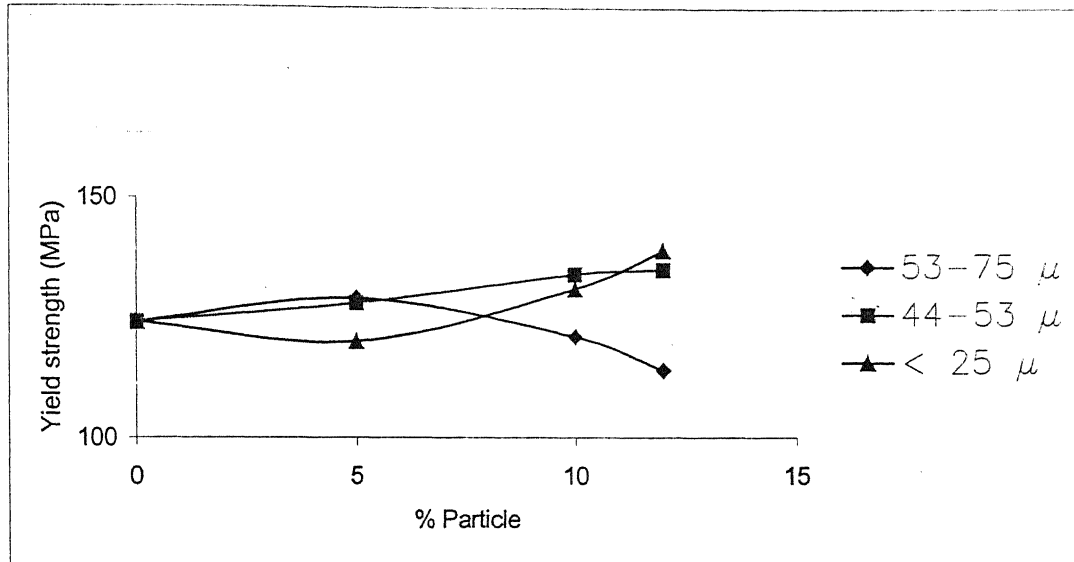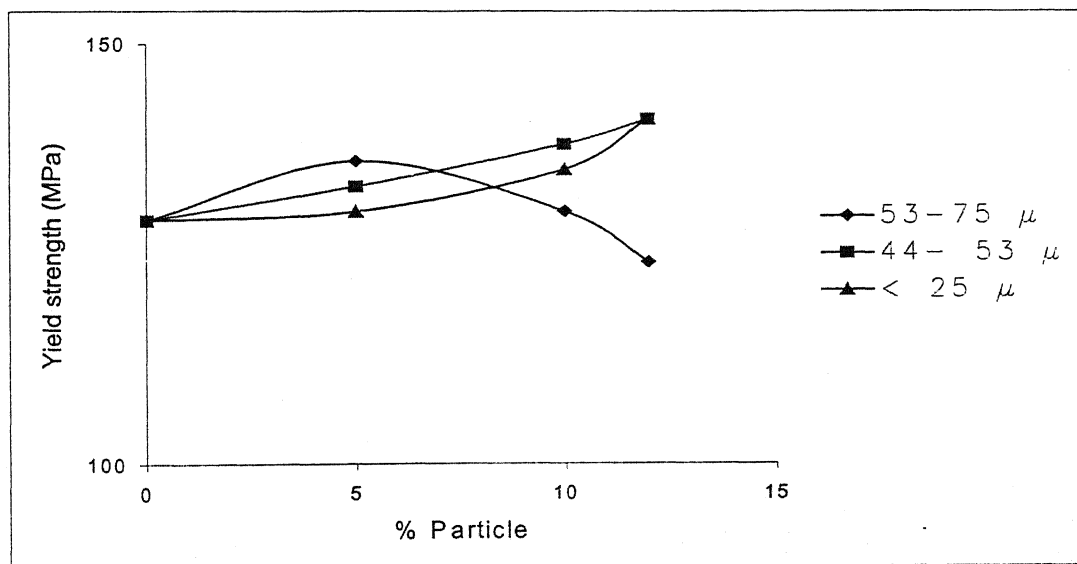**Fig. 4.13 :** **Schematic diagram of yield strength (T) at different**
wt. fraction of particles (rpm of drum caster-16)

Comparing the strength of the composites (<25 μm particle size) of the present investigation with commercially available composites, shown in Fig. 2.5, it can be deduced that gain in strength of composite from its matrix alloy is better for the strip obtained by SRCSC method.

As shown in Fig.4.14 the elongation of the composite is strongly affected by reinforcing parameters. Higher percentage of particle reduces the ductility, due to reduced mean spacing between particles.

Hardness measurements were performed on the Rockwell hardness tester using 'B' scale. As shown in Fig. 4.15 the hardness exhibits a similar trend as of the tensile strength with respect to particle size. Similar explanation as given above for increased tensile strength will also be valid for the increased hardness at higher percentage and for smaller size particles.

**Fig. 4.14 : Schematic diagram of % elongation at different
wt. fraction of particle (rpm of drum caster-16)**

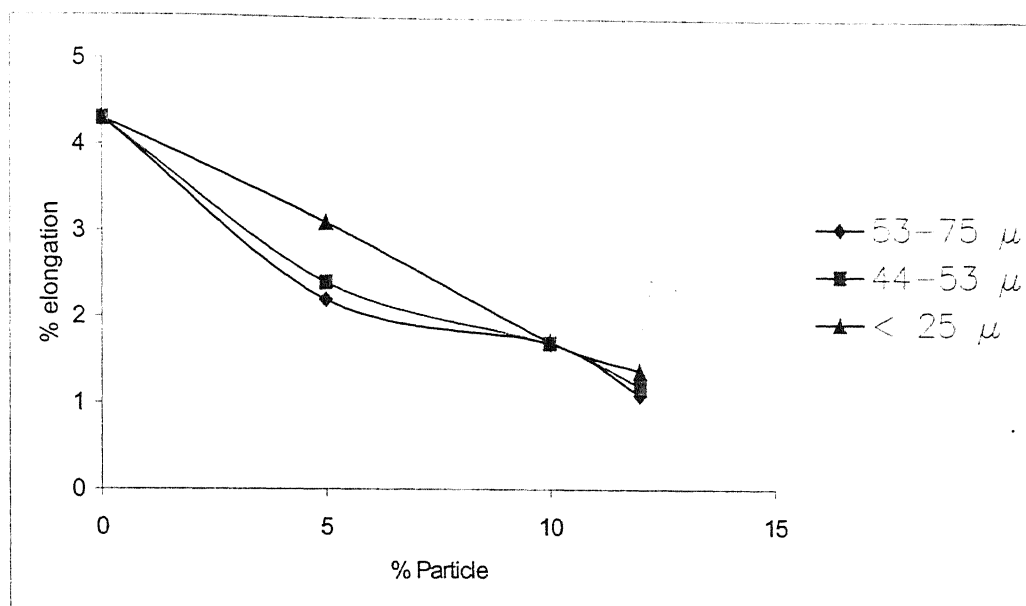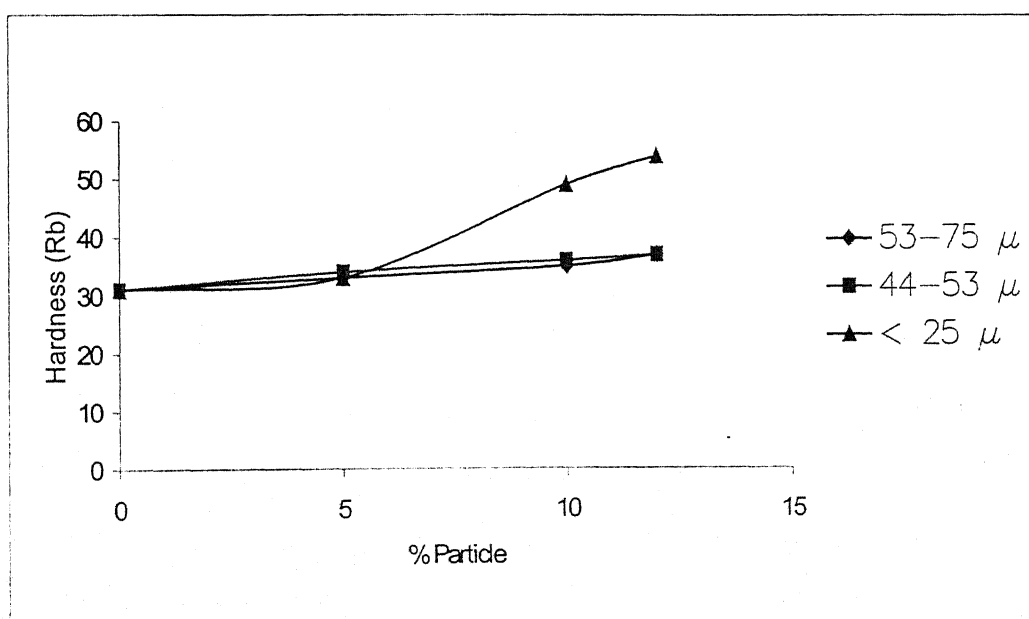**Fig.4.15: Schematic diagram of Hardness (Rockwell B) with
different wt. fraction particle (rpm of drum caster-16)**

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4.4.2 Effect of speed of rotation of caster drum:

Speed of the rotation of the caster drum is the one of the most influencing factors regarding the properties of the strip. Table 4.2 shows the effect of speed of rotation of caster drum on the tensile strength of the strip.

It is seen that the ultimate tensile strength increases when the speed of rotation is changed from 12RPM to 16RPM. The reason for increase in strength at higher speeds can be attributed to the increased rate of solidification, which, in turn, affects the grain size.

Both, the increased mechanical contact between the drum and the strip and the enhanced heat transfer coefficient because of the higher rate of heat withdrawal; produce a product with fine grain structure at higher rotational drum speeds [53]. Increase in strength is also due to finer eutectic structure obtained at higher rate of solidification. This is further influenced due to less clustering and agglomeration of particle at higher RPM. For the transverse direction also similar trends are observed. The reduction in the ductility of strips at higher speed of rotation is partly due to the increased porosity of the strips. The hardness increases with increased RPM of the drum.

Table 4.2: Effect of RPM of drum caster (wt. fraction of particle-10%)

Size of Particle	RPM of Drum Caster	Tensile Strength (MPa) L / T	Yield Strength (MPa) L / T	% Elongation	Hardness (Rockwell B)
44-53 μm	16	154/159	134/138	1.7	49
	12	149/153	122/126	2.1	45
< 25μm	16	182/188	131/135	1.72	49
	12	172/177	126/130	2.31	45
Al-Si alloy	16	148/154	124/129	4.3	31
	12	140/146	119/123	4.5	28

4.4.3 Elevated temperature tensile properties:

Elevated temperature tensile strength and % elongation is shown in Fig. 4.16 and Fig. 4.17, respectively. From Fig.4.16 it can be inferred that high strength of the composite is retained at elevated temperatures. A sharp decrease in strength of the composite as compared to alloy can be seen above 250°C. The reason behind this behavior of composite is explained below.

As it has been discussed in section 2.3.1, strengthening of composite at room temperature is due to:

- the presence of SiC hard particles in softer matrix, which hinder dislocation motion and thus increase strength,

- the difference in CTE between two phases produces thermal mismatch at the interface. High density of dislocation and the particles act as obstacles to dislocation movement and result in increased strength.

But at elevated temperatures matrix deformation plays a key role in strengthening of the strips. As stated above and also seen in Fig. 4.16, that at higher temperatures (>= 250 °C), when the matrix becomes soft the particles may debunk with the matrix, leaving high density of voids. This void formation ultimately acts so as to reduce the strength. Thus at higher temperatures (>=250 °C) reinforcing particles reduce the strength of composite even below the strength of the alloy matrix by additional voids formation [54].

As is evident in Fig.4.17, the % elongation is quite good and it indicates the high deformability of the strip at elevated temperatures.
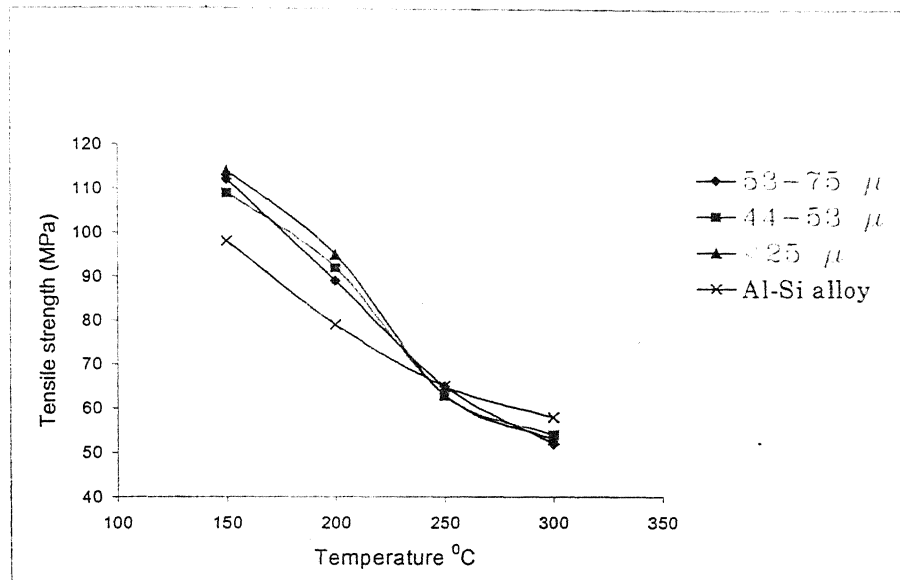
Fig. 4.16 : Schematic diagram of elevated temperature tensile strength (% of particle-10, rpm of drum caster-16)
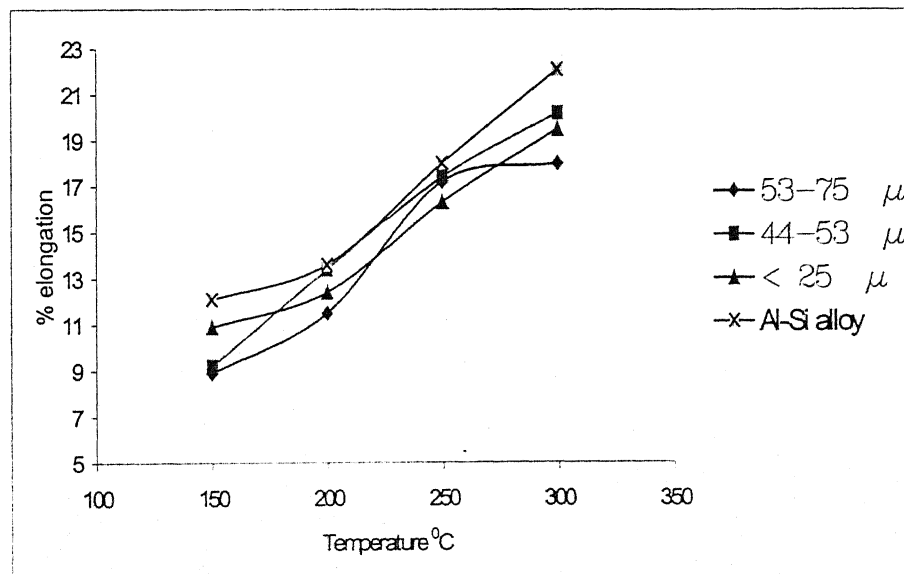

Fig. 4.17 : Schematic diagram of % Elongation at elevated temperature (% of particle-10, rpm of drum caster-16)

CHAPTER 5
Summary and Conclusions

The present investigation involves two parts. In the first part, composite strips of the aluminum reinforced with SiC particles have been produced using single roll continuous strip caster (SRCSC)-designed and developed by Mehrotra and coworkers for producing near-net-shape metal strips. Small percentage of magnesium is added to the Al alloy melt, to improve wetting characteristic the refractory reinforcement particulate by the matrix. Particles are preheated before their incorporation into the melt. The melt is continuously stirred to further improve the wetting characteristics of the particles with the melt. Vortex method has been used for preparing the composite melt.

In the second part, evaluation of microstructure and mechanical properties has been carried out. Microstructural investigations were based on optical and electron microscopy to study the distribution of particles in the cast strips and internal quality of the strips. Optical microscope is used to calculate recovery of particle by quantitative metallography. Mechanical properties such as strength, ductility, hardness and elevated temperature strength are examined for the composite strips produced at various operating conditions such as reinforcement particle size and percentage of particle, speed of rotation of the caster drum, etc. Effect of these parameters on the surface quality of the composite strips has also been examined. Operating parameters are optimized to obtain optimum properties.

From the analysis of the experimental data, the following conclusions are drawn:
- It is Possible to produce particle reinforced MMCs strips by vortex method combined with Single Roll Continuous Strip Casting Technology.
- Size of the reinforcement particles directly affects the mechanical properties and distribution of particle in the matrix.
- Smaller size ($<25\mu m$) particles improve the strength to a greater extent.
- Larger size ($> 40\mu m$) particles show better distribution and recovery.
- Tendency of particles to segregate increases as the size decreases.
- The roll side surface of the strip is smooth as compared to the topside surface.

➢ Increase in rpm of the drum caster gives better distribution of particles and increases the strength.

➢ Hardness shows the similar trends as of the strength.

➢ Composite strip shows better strength at elevated temperature ($<250^0C$) than the matrix alloy.

➢ Ductility of the composite decreases with increase in wt. Percentage of the particles.

➢ Using the optimum value of the process parameter in the different stages of this process technology a reasonably good combination of properties can be achieved.

APPENDIX A1

Physio-chemistry of Wetting:

The wettability of a solid by a liquid melt is indicated by the contact angle θ. This angle is correlated to the three surface energy γ_{sg}, γ_{lg}, γ_{sl}, of solid-gas, liquid-gas and solid-liquid interfaces as shown in Fig. respectively through the well-known Young's equation.

$$\gamma_{lg} \cos\theta = \gamma_{sg} - \gamma_{sl}$$
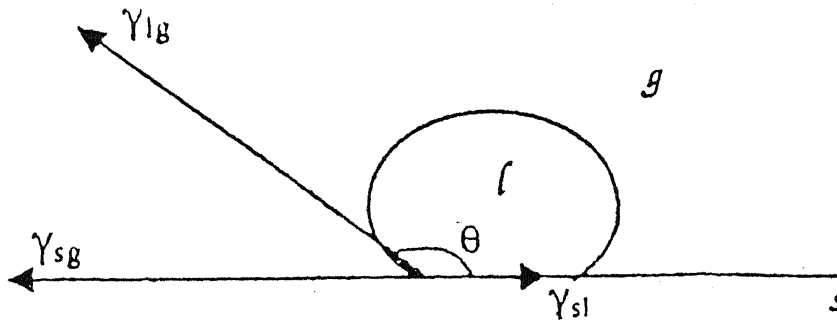
Fig. : Schematic diagram of wetting principle

For the wetting to take place, the necessary condition is:

$$\cos\theta > 0$$

i.e. $\theta < 90^0$

From this condition it is inferred that lower the angle of contact, better is the wetting. In other words

$\gamma_{sg} > \gamma_{si}$

The driving force Fw, for wetting can be defined as

$Fw = \gamma_{sg} - \gamma_{sl}$

In the extreme case when

$F_w >= \gamma_{lg}$; $\theta = 0^0$

the liquid spreads spontaneously on the solid. For contact angle $\theta > 90^0$, the capillary effect requires an external pressure in order that the liquid wets the solid in contact with it. However, the application of pressure does not always completely solve the problem, since shrinkage during the solidification may be large enough to cause debonding or void formation.

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